









## THE TECHNOLOGY OF SUGAR



# THE TECHNOLOGY OF SUGAR

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<i>First Edition issued</i>	. . . . .	<i>October, 1903</i>
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## PREFACE TO THE FIRST EDITION

THE Author would have liked to have dealt with the cultivation of the sugar beet, the influence of pedigree seed on the sugar content of the beet, and to have described more fully the culture of the sugar cane; but he deemed it better to devote the bulk of the space at his disposal to the wants of the Sugar-works Chemist, the Engineer, and the Public Analyst. Sugar-cane and sugar-beet cultivation are, in fact, separate industries from sugar manufacture and refining. He trusts that the book may be useful not only to those for whom it is primarily intended, but to all those who have to do with the subject; *e.g.*, Excise and Custom Officers, and all intelligent members of society who may wish to know something more about sugar. Now that the Bounty system is about to be abolished, the British sugar manufacturer must look to his laurels. The Bounty system, however prejudicial, was not the only cause of the decline and fall of sugar refining in Britain and sugar manufacture in British Colonies. Obsolete machinery and methods contributed much to the decadence of the industry.

As the book is not intended for the purely theoretical chemist, therefore in their present incomplete state of transition it has not been thought necessary to enter either into such questions as the synthesis of sugars, or to discuss the newer theories and classification of saccharine bodies. As to the chemical nomenclature used, the Author has exercised his right to use what seemed to him best suited for the occasion. But when will theoretical reviewers realise that *nomenclature* is not *chemistry*, and that a man may be a sound chemist whatever system he uses, and that it does not necessarily follow that if he uses one system he is ignorant of the other? The book has not been burdened with voluminous tables of imports of sugar and other statistics of only ephemeral interest, nor

has it been made a record of inventions of machinery and processes. Those interested can get all the abridgments of sugar-patent specifications *ab initio* for a nominal sum, and the statistics in regard to sugar are to be found for the preceding five years in the *Annual Statement of Trade* prepared by H.M. Customs.

The Sugar-works Engineer and Chemist will find it to his advantage to supplement this treatise with Hausbrand's *Evaporating, Condensing, and Cooling Apparatus* (published by the same firm as this treatise), which is a perfect storehouse of information in regard to everything pertaining to evaporation and the thermo-dynamical laws by which it is regulated.

The Author's task has in many instances been limited to assimilating the works of his predecessors: Ure, Wallace, Brande, Scoffern, Newlands, Muspratt, and Gill, in Britain; Horsin-Deon, Sidersky, and Cambier, in France; Wiley and Spencer, in America; Jelinek and others, in Austria; Claassen and Hausbrand, in Germany. To each of these now alive, and the other living writers to whom the Author is indebted, he returns his heartfelt thanks; but the special literature of this subject is very voluminous, and much of it does not admit of useful condensation.

We all hope for a speedy revival of the Sugar Industry both at home and in our Colonies; and should this volume assist in some measure, it will not have been written in vain.

J. G. M.

LONDON, October, 1903.

## PREFACE TO THE SECOND EDITION

It seldom falls to the lot of the Author of a Technical Treatise to be called upon so soon to write a Preface to a Second Edition. The phenomenal success of the First Edition is gratifying alike to Publishers and Author. Both have done their best to improve the New Edition in every way. The illustrations, tabular data, and reading matter have all been added to and extended very considerably. It is hoped the book will thus be rendered worthy to follow its predecessor.

J. G. M.

LONDON, *January*, 1906.





## TABLE OF CONTENTS

	PAGE
BIBLIOGRAPHICAL NOTE . . . . .	iv
PREFACE TO FIRST EDITION . . . . .	v
PREFACE TO SECOND EDITION . . . . .	vii
TABLE OF CONTENTS . . . . .	ix
LIST OF ILLUSTRATIONS . . . . .	xix
LIST OF TABLES . . . . .	xxiii

### SECTION I.

#### BEET SUGAR.

##### CHAPTER I.

##### COST OF PRODUCTION—VALUATION—PURCHASE AND PRELIMINARY TREATMENT OF BEETS.

	PAGE
Beet-sugar Prospects in Great Britain . . . . .	1
Cost of Growing an Acre of Sugar Beets . . . . .	2
Weight of Crop per Acre . . . . .	2
Purchase and Analysis of Beets . . . . .	7
Heading and Tailing . . . . .	8
Saccharimetrical Tables . . . . .	9
Preliminary Treatment of the Beets . . . . .	24
Washing and Weighing . . . . .	25

##### CHAPTER II.

##### DIFFUSION.

Diffusion—Beet Slicers—Knives—Diffusion Batteries—Pulp Eliminators —The Chemical Control of Diffusion—The Feeding Value of Ex- hausted Pulp . . . . .	33
---	----

## TABLE OF CONTENTS

## CHAPTER III.

## CARBONATATION AND FILTRATION.

	PAGE
Pectic and Albumenoid Impurities of Beet Juice . . . . .	69
Defecating Agents—	
Lime . . . . .	78
Baryta . . . . .	82
Strontia . . . . .	82
Horsin-Deon's and Gibert's Continuous Systems . . . . .	90
Manufacture of Carbonic Acid and Quicklime . . . . .	95
Chemical Analysis and Titration of the Limed Juice . . . . .	97
The Chemical Control of the Carbonation Process . . . . .	97
The Exhaustion and Utilisation of the Sludge . . . . .	105
Manurial Value of Sludge . . . . .	107
Composition . . . . .	107
Estimation of Sugar lost in Sludge . . . . .	108
Filtration—	
Filter Presses . . . . .	102
Charcoal Filters . . . . .	109
Bag Filters . . . . .	119
Mechanical Filters . . . . .	120
Manufacture, Washing and Revivifying of Animal Charcoal . . . . .	111
Disposal of Spent Char . . . . .	118

## CHAPTER IV.

CONCENTRATION OF BEET JUICE TO SYRUP IN MULTIPLE-EFFECT  
EVAPORATION VESSELS (IN VACUO).

Differentiation between Evaporation and Distillation, Costliness and Wasteful Nature of Open Pan Evaporation over Direct Fire . . . . .	124
The Old Open Pan and Fire Heat <i>versus</i> the Newer Vacuum Pan and Steam Heat . . . . .	126
Comparative Economy of Evaporation by Single and Multiple-effect . . . . .	137
Thermo-dynamical Data and Formulæ and their Practical Application . . . . .	139
Water Evaporated per Square Metre of Heating Surface in Double-effect . . . . .	145
Water Evaporated by Triple-effect in Factory Working 10 Metric Tons of Beets per hour . . . . .	145
Method of Working Triple-effect Plant . . . . .	156
Froth Preventers and Sugar Recoverers . . . . .	159
Condenser and Air-pumps . . . . .	161
The Pressure in the Condenser . . . . .	165
Quadruple and Quintuple Evaporation . . . . .	166
Utilisation of the Multiple-effect Steam in Reheating the Juice, and the Saving thus effected . . . . .	169
Diameter of Pipe from Exhaust Steam Reservoir to No. 1, from No. 1 to No. 2, from No. 2 to No. 3, for Triple-effect of different ca- pacities . . . . .	171

# TABLE OF CONTENTS

xi

	PAGE
Fuel Consumption in Austrian Factories . . . . .	172
The Ammoniacal Vapours and Pumps . . . . .	172
Elimination of Ammoniacal Gases . . . . .	173
Cleaning the Apparatus . . . . .	174
Removing Encrustation . . . . .	174
Horizontal Systems of Multiple-effect Evaporation . . . . .	174
The Yaryan Streaming Process . . . . .	175
Automatic Sampling of Concentrated Juice (Syrup) . . . . .	176

## CHAPTER V.

### BOILING DOWN OF THE BEET SYRUP TO CRYSTALLISING-POINT IN VACUUM PANS—CRYSTALLISATION IN MOTION—THE TREATMENT OF WEAK LIQUORS AND OF LIQUORS OF BAD QUALITY.

Degree Baumé of Syrup . . . . .	177
Preliminary Filtration . . . . .	178
Evaporation in Vacuum Pans—	
Horizontal . . . . .	179
Vertical . . . . .	181
The Point of Saturation of Sugar Solutions at various Temperatures . . . . .	180
Calandria . . . . .	182
Steam Pipes of . . . . .	182
Coils . . . . .	183
The Radiating Heater . . . . .	183
Boiling in Grain . . . . .	184
Crystallisation in Motion . . . . .	185
The New <i>Masse Cuite</i> Receivers (Coolers for) . . . . .	187
Sugar Boxes . . . . .	190
The Old Storing and Cooling Tanks . . . . .	190
Difficulties in Boiling Juice from Spoilt Beets . . . . .	192
Analyses of Syrup and <i>Masses Cuites</i> (Scheme for) . . . . .	194
Composition of <i>Masses Cuites</i> according to Density . . . . .	197
Composition of <i>Masses Cuites</i> and Syrups according to Density . . . . .	198
Total Steam consumed in each Operation per Ton of Beets . . . . .	199
Total Steam Expended by all Engines and by each Engine in a Beet-sugar Works . . . . .	200
Steam consumed per Hectolitre of Juice . . . . .	200
Degree Baumé of Syrup at Exit from Triple-effect . . . . .	201
Syrup required per Hectolitre of <i>Masse Cuite</i> . . . . .	201
Water to Evaporate from Syrup to get Hectolitre of <i>Masse Cuite</i> . . . . .	201
Water Evaporated per Hectolitre of Syrup to Transform into <i>Masse Cuite</i> . . . . .	201
<i>Masse Cuite</i> obtained per Hectolitre of Syrup . . . . .	201

## TABLE OF CONTENTS

## CHAPTER VI.

TURBINING OF BEET-SUGAR *MASSÉS CUITES*.

	PAGE
Magnitude of Centrifugal Force of Turbines . . . . .	202
Method of Working . . . . .	203
Acceleration of Speed in Driving Turbines . . . . .	203
Speed with different Grades of Sugar . . . . .	204
Over-driven Turbines . . . . .	204
Suspended Turbines (Weston's) . . . . .	204
Under-driven Turbines . . . . .	204
Water-driven Turbines . . . . .	204
Continuous Turbines . . . . .	206
Plan of Turbine Room . . . . .	206
Charging Turbines, Old Routine . . . . .	207
Recent and more or less Automatic Methods . . . . .	207
Concrete Sugar Truck . . . . .	208
Overhead Feeding Sugar Truck . . . . .	209
Wet Sugar Elevator . . . . .	211
Dry Sugar Elevator . . . . .	211
Air Compressor for Discharging Sugar Trucks . . . . .	211
Cleansing the Sugar by Injection of Steam and Air . . . . .	212
Cleansing to get very White and Ordinary Yellow Sugars . . . . .	213
"Firsts" Sugars and "Firsts" Syrups . . . . .	214
Molasses Cutters . . . . .	214
"Seconds" . . . . .	215
"Thirds" . . . . .	216
Fill-houses . . . . .	216
Influence of Purity of <i>Masse Cuite</i> on Yield of Sugar . . . . .	217
Crystallisation in Motion—How far Practicable to Push with Low Quality Products . . . . .	218
Transferring Turbined Sugar to Granary . . . . .	219
Drying, Sifting, Mixing, Screening, and Bagging-up Turbined Sugar . . . . .	220
Automatic Mixer of Turbined Sugar . . . . .	221
Automatic Bagging-up and Weighing Machine . . . . .	223
Beet-sugar Warehouses . . . . .	223

## CHAPTER VII.

EXTRACTION OF ALL AVAILABLE SUGAR FROM BEET-SUGAR  
MOLASSES.

Beet-sugar Molasses, its Composition and Nature . . . . .	224
Extraction of Sugar from Molasses . . . . .	224
The Old Alcoholic Method . . . . .	225
Dubrunfant's Osmogène (Dialysing Method) . . . . .	226
Evaporating Osmogènes . . . . .	226
Sucrate of Lime Process (Steffen's) . . . . .	227

## TABLE OF CONTENTS

xiii

	PAGE
Manoury's Alcoholic Method . . . . .	229
The Strontia Method . . . . .	230
Advantages of Sucrate of Strontia over Sucrate of Lime . . . . .	230
Continuous Sulphitation of the Syrup and Drainage Liquors . . . . .	230
Attempts to Abolish Molasses . . . . .	232
Crystallisation in Motion . . . . .	232
Steffen's Process . . . . .	233

## SECTION II.

### CANE SUGAR.

#### CHAPTER VIII.

##### THE SUGAR CANE AND ITS CULTIVATION—EXTRACTING THE JUICE BY MILLING.

The Sugar Cane and its Average Richness in Sugar . . . . .	236
The Composition of the Sugar Cane . . . . .	236
Colouring Principles . . . . .	238
Cane-sugar Wax or Cerosin . . . . .	238
Valuation of . . . . .	238
Culture of the Sugar Cane . . . . .	239
Cane Conveyors . . . . .	240
Cost and Yield per Acre, Profit and Loss . . . . .	241
The Yield of Sugar per Ton of Cane . . . . .	241
Extraction of the Juice by Milling . . . . .	242
By Double Milling after Immersion . . . . .	243
Modern Mills . . . . .	243
The Mechanics of the Cane Mill . . . . .	246
Speed of Rolls . . . . .	247
Machine for Forcing the Juice from the Cane by Percussion . . . . .	248
Shredders . . . . .	249
Perichon's Systematic Levigation of Bagasse . . . . .	250
Bagasse as Fuel . . . . .	253
Furnace for Burning Green Bagasse . . . . .	253

#### CHAPTER IX.

##### SUGAR-CANE DIFFUSION—THE RESULTS OBTAINED ON THE LARGE SCALE BY THE UNITED STATES DEPARTMENT OF AGRICULTURE AT MAGNOLIA, LOUISIANA, U.S.A. (AN ELABORATE REPORT).

Coal Consumption in Fuel <i>versus</i> Milling . . . . .	254
Cane-sugar Diffusers, Elevators, Feed-chute and Calorisators . . . . .	255
Cane Cutter (Diffusion) . . . . .	257

## TABLE OF CONTENTS

	PAGE
Cane Shredder (Diffusion) . . . . .	259
Dimension of Cells . . . . .	259
Dimension of Calorisators . . . . .	260
Juice and Water Mains . . . . .	260
Circular Batteries <i>versus</i> Line Batteries . . . . .	260
Manipulation of Battery . . . . .	263
Influence of Size and Shape of Cell . . . . .	264
Clarification by Liming in Cell . . . . .	265
Working Temperature . . . . .	266
Comparative Temperature in Sugar-cane and Sugar-beet Diffusion Cells . . . . .	267
Dilution . . . . .	269
Cane, per Cell . . . . .	269
Cane, per Cubic Foot of Cell Capacity . . . . .	269
Diffusion Juice Drawn, per 100 Lb. Cane . . . . .	269
Actual Dilution . . . . .	269
Apparent Dilution . . . . .	269
Mean Composition of Normal and Diffusion Juices, Density, Sucrose per cent., Reducing Sugars per cent., Coefficient of Purity, Glucose per cent., Inversion per cent. . . . .	269
Increase in Diffusion Juice Evaporation over Milling . . . . .	269
Fuel Consumption in Beet <i>versus</i> Cane Diffusion . . . . .	270
Crop Report—	
Firsts . . . . .	271
Seconds . . . . .	271
Thirds . . . . .	271
Fourths . . . . .	271
Available Sucrose, Lb. per Ton of Juice . . . . .	272
Sucrose Obtained, Lb. per Ton of Juice . . . . .	272
Relation between Density of Sugar Cane Juice and Richness in Sugar . . . . .	274

## CHAPTER X.

## DEFECATION AND EVAPORATION OF CANE JUICE.

Defecation of the Cane Juice . . . . .	275
Cane Juice and its Properties . . . . .	276
Eliminating Impurities . . . . .	277
Sulphitation . . . . .	278
Evaporation to Crystallisation-point in Vacuum Pans . . . . .	279
The Old Wetzell's Pan and Chenailier's Pan for Converting Juice into Syrup . . . . .	281
The New Triple, Quadruple-effect for Converting Juice into Syrup . . . . .	281
The Old Howard's Pan for Boiling Syrup to Strike-point . . . . .	281
Boiling to Strike-point, Turbining, etc., as in Case of Beets . . . . .	281
Molasses . . . . .	281
Molascuit . . . . .	281
Compact Plant for Colonial Use . . . . .	282

# TABLE OF CONTENTS

ix

## SECTION III.

### CHAPTER XI.

#### SUGAR REFINING.

	PAGE
The Raw Material . . . . .	283
White Sugar . . . . .	283
Yellow Sugar . . . . .	283
Brown Sugar . . . . .	284
Purchase and Selection . . . . .	284
Typical Analyses of different Brands of Raw Sugar . . . . .	284
Blending Prior to Refining . . . . .	285
A Product of Predetermined Quality to be aimed at and Work regulated accordingly . . . . .	285
Preliminary Turbining . . . . .	286
Solution by Water and Steam . . . . .	288
Defecation . . . . .	290
Filtration . . . . .	291
Taylor's Filters . . . . .	291
Animal-charcoal Filters . . . . .	294
Selection of Good Quality Char . . . . .	295
Function of the Nitrogen . . . . .	296
Injurious Action of Free Ammonia and Ammonium Sulphide in Char . . . . .	296
Presence of Interstitial Occluded Gases in Animal Charcoal . . . . .	297
Substitutes—Basic Acetate of Lead and Sulphurous Acid . . . . .	297
Decolorisation by Ozone . . . . .	298
Defecation of Cane Sugar by the Double-carbonatation Process . . . . .	298
Size or Grist of the Granulated Char . . . . .	299
Amount of Charcoal to Decolorise Given Weight of Sugar . . . . .	299
Loss of Sugar in the Bone-black Room . . . . .	300
Water Used in Refining 100 Tons of Sugar . . . . .	300
Revivifying Animal Charcoal . . . . .	302
Change in Composition on repeated Re-burning and Re-use of Animal Charcoal . . . . .	303
Method of Removal of Mineral Salts . . . . .	304
Bad Effect of permanently Hard Water . . . . .	304
Old and New Charcoal, Ratio of Bulk to Gravity . . . . .	305
Amount of Char per Ton of Raw Sugar . . . . .	305
Soft Water begets Iron Salts in the Syrup . . . . .	307
Treatment of the Charcoal with Dry Hydrochloric Acid . . . . .	307
Boiling the Sugar in Vacuum Pan . . . . .	308
Boiling-point . . . . .	308
Howard's Vacuum Pan . . . . .	309
The Steam Jacket . . . . .	309
Coil, Barometers, Thermometers and Feeding Apertures . . . . .	309
The Wet Air-pump . . . . .	311



## TABLE OF CONTENTS

	PAGE
Injection Condenser . . . . .	318
Instructions for using the Vacuum Pan . . . . .	313
Testing a Vacuum Pan and Air-pump for Tightness . . . . .	315
Precautions in Applying Steam . . . . .	315
Heating Power of High and Low Pressure Steam . . . . .	317
Detecting Leakage . . . . .	318
Measuring the Liquor . . . . .	319
The Capacity of the Condensers . . . . .	319
Decrepitation in Pan as Crystals begin to Form . . . . .	320
<i>The Flash</i> . . . . .	321
<i>Taking the Proof</i> . . . . .	321
Examination of the Proof . . . . .	321
Feeding the Pan . . . . .	322
Final Precautions . . . . .	322
Boiling by Doubles . . . . .	324
Boiling by the Thread Proof . . . . .	324
Cleansing of the Proof-stick . . . . .	325
Reheaters . . . . .	325
The Fill-house . . . . .	329
The Making of Sugar-loaves . . . . .	330
Liquoring . . . . .	331
"Magma Liquor" . . . . .	332
Fine Liquor . . . . .	332
The Facing Machine . . . . .	333
Facilitating Percolation and Drainage by Suction . . . . .	333
Turbining Cube-sugar Tablets . . . . .	334
Raw Syrup or Molasses . . . . .	335
Manufacture of Caramel . . . . .	335

## SECTION IV.

## CHAPTER XII.

THE CHEMISTRY OF SUGARS—ANALYSIS OF COMMERCIAL SUGARS  
AND OF MERCHANDISE, ETC., CONTAINING SUGARS.

Definition and Classification of Sugars, Monoses, Bioses and Trioses . . . . .	337
The Chemistry of Sucrose, Lactose, Maltose, Glucose and Levulose, and their Saccharimetric, Volumetric and Gravimetric Determination . . . . .	340
Sucrose, Cane Sugar, Density of Solutions . . . . .	339
Sucrose in Pounds, in 1 Gallon of Syrup of Consecutive Densities . . . . .	341
Glucose . . . . .	345
Levulose . . . . .	349
Maltose . . . . .	350
Saccharimetry . . . . .	353
Volumetric Analysis of Sugars . . . . .	364

# TABLE OF CONTENTS

xvii

	PAGE
Gravimetric Analysis of Sugars . . . . .	380
The Analyses of Raw Sugars . . . . .	387
Estimated Yield of Raw Sugar in Refined . . . . .	395
The Chemistry of Molasses, Invert Sugar and Raffinose . . . . .	395
Clerget's Table . . . . .	400
Estimation of Raffinose . . . . .	402
Composition of Table Syrups . . . . .	403
Official German Methods used in Taxing Sugars . . . . .	404
Chocolate and Candies . . . . .	412
Raffinade and Santonin Wafers . . . . .	414
Cakes, Bonbons and Marzipan . . . . .	414
Sugar-coated Fruits, Marmalades and Jellies . . . . .	415
Liquors . . . . .	416
Invert Cane Sugar . . . . .	417
Commercial Glucose, Solid . . . . .	418
Commercial Glucose, Syrup . . . . .	418
The Estimation of Sugar in Wine . . . . .	419
Milk . . . . .	421
Honey . . . . .	423
Diabetic Urine . . . . .	423
Jam . . . . .	424
Bananas . . . . .	425
City Guilds Examinations . . . . .	427
INDEX . . . . .	429



## LIST OF ILLUSTRATIONS

FIG.	PAGE
1. Pellet's Beet Pulper . . . . .	15
2. Drill for Sampling Beets . . . . .	15
3. Hanriot's Beet Pulper . . . . .	16
4. Apparatus for Continuous Saccharimetrical Analysis . . . . .	19
5. Fricourt's Machine for Washing Beets . . . . .	26
6. Stone-eliminator . . . . .	27
7. Beet-wiping Machine . . . . .	28
8. Beet Shaker . . . . .	29
9. Beet Elevator . . . . .	31
10. Circular Diffusion Battery . . . . .	38
11. Slicing Knife and Knife-holder . . . . .	43
12. Knife-holder, Non-stone Remover . . . . .	43
13. Knife-holder and Stone Remover . . . . .	44
14. Knife-holder . . . . .	44
15. Fontane and Rasmann's Slicer . . . . .	45
16. Rotary Drum Slicer . . . . .	46
17. " " on old body . . . . .	47
18. " " ordinary body . . . . .	48
19. " " " " . . . . .	49
20. Slice-sampling Apparatus . . . . .	50
21. Circular Diffusion Battery . . . . .	51
22. Feeding Diffusion Batteries . . . . .	52
23. Feeding and Clearing Circular Battery . . . . .	54
24. Cail's Diffuser . . . . .	55
25. Air-removing Apparatus . . . . .	56
26. Bergneen's Exhausted Pulp Press . . . . .	59
27. Pfeiffer's Automatic Diffusion Process . . . . .	66
28. Pelhardt's Pulp Eliminator . . . . .	68
29. Beet Juice Liming Vessels . . . . .	73
30. Intermittent Carbonatation Tanks . . . . .	80
31. Horsin-Deon's Automatic Register . . . . .	84
32. Automatic Juice-sampling Apparatus . . . . .	86
33. Langen's Automatic Register . . . . .	87



## XXI

	PAGE
79. Sugar Sifter . . . . .	220
74. Automatic Conveyance of Turbined Sugar to Warehouse Bins, Core Crushing Rolls, Helical Mixer, and Automatic Bagging-up and Weighing Machine . . . . .	221
75. Refrigerator Mixer used in Molasses Separation Process . . . . .	229
76. Quarez's Continuous Sulphitation of Molasses . . . . .	231
77. " " " " " " . . . . .	231
78. The Sugar Cane . . . . .	237
79. Cane Conveyor . . . . .	240
80. Sugar-cane Mills . . . . .	243
81. Milling and Remilling . . . . .	244
82. " " " " " " . . . . .	244
83. Cane Shredder . . . . .	245
84. Cane-mill Rollers . . . . .	245
85. Friction Wheels . . . . .	246
86. Bessemer's Cane-pressing Machine . . . . .	249
87. Perichon's Bagasse-levigation Process . . . . .	250
88. Furnace for Burning Green Bagasse . . . . .	253
89. Cane-sugar Diffusers . . . . .	255
90. Cane Cutter . . . . .	258
91. " " " " " " . . . . .	259
92. Cambray's Sulphur Battery . . . . .	278
93. " " Sulphitation Pan . . . . .	280
94. Compact Cane-sugar Plant . . . . .	282
95. Battery of French Turbines with Automatic Conveyor of Turbined Sugar to Mixer and Bagging-up Machine . . . . .	287
96. Taylor's Filter Bags . . . . .	293
97. Howard's Vacuum Pan . . . . .	310
98. Proof-stick and Accessories . . . . .	310
99. Vacuum Pump . . . . .	312
100. Vacuum Pan . . . . .	326
101. Diagrammatic Representation of Sugar-refining Plant . . . . .	328
102. Vacuum Pans and Reheater . . . . .	329
103. Sugar-loaf Moulds for Draining Loaves by Suction . . . . .	330
104 and 105. Sugar-tablet Mould and Turbine for Cube Sugar . . . . .	334
106. Haensch's Half-shadow Saccharimeter . . . . .	356
107. Polarisation Discs . . . . .	357
108. Zeiss's Portable Saccharimeter . . . . .	359



# LIST OF TABLES

## SECTION I.

### BEET SUGAR.

#### A. TABLES RELATING TO COST OF GROWING BEETS.

	PAGE
I. Cost of Growing a Hectare of Beets in the Plain of Laon, France . . . . .	3
II. Cost of Growing Beets in the United States . . . . .	4
III. Actual Cost of Growing Beets in U.S.A. . . . .	4
IV. Cost of Growing Beets—a French Estimate . . . . .	5
IV.A. Return on Capital Invested . . . . .	6

#### B. TABLES RELATING TO TESTING OF BEETS AND BEET JUICE.

V. Corrections to bring Density of Beet Juice at Various Temperatures to Density at 15° C. . . . .	9
VI. Estimation of the Amount of Sugar in Beetroots (Edouard Delville) . . . . .	11
VII. Density of Solutions of Sucrose in Water at 15° C. (Scheibler) . . . . .	12
VIII. Expenses incidental to Testing Beets for Seed-producing Purposes . . . . .	20
IX. Correction Table for Increase of Volume by Addition of Basic Acetate of Lead . . . . .	21
X. Table for the Analysis of Sugar Beets. French Saccharimeter (F. Sachs) . . . . .	22
XI. Table for the Analysis of Sugar Beets. German Saccharimeter (F. Sachs) . . . . .	23
XII. Analysis of Beet Juice. Comparison between Optical and Chemical Methods . . . . .	24



## LIST OF TABLES

## C. TABLES RELATING TO TREATMENT OF BEET JUICE AND VALUATION OF RESIDUALS.

	PAGE
XIII. Analyses showing Nutrition Value of Beet Diffusion Pulp (Residual) . . . . .	60
XIV. Analyses of Weak Liquors (Scheibler's Table) . . . . .	62
XV. For the Analyses of Weak Sugar Liquors. (French Saccharimeter. Normal weight, 16.29 grammes.) . . . . .	63
XVI. Exhausted Pulp.—(Juice of exhausted pulp. 400-millimetre tube. Normal weight of instrument, 16.2 grammes; addition of $\frac{1}{10}$ basic acetate of lead.) . . . . .	64
XVII. Exhausted Pulp.—(Table for German instruments. Juice of exhausted pulp. 400-millimetre tube. Normal weight of instrument, 26.048 grammes; addition of $\frac{1}{10}$ basic acetate of lead.) . . . . .	65
XVIII. Solubility in Sugar Solutions of certain Insoluble Substances (Jacobsthal) . . . . .	70
XIX. Composition of the Sugar Beet and its Juice . . . . .	71
XX. Amount of Lime Absorbed by Syrups of Different Densities with Densities of Resulting Syrups and their Percentage Composition (Peligot) . . . . .	75
XXI. Percentage of Quicklime (CaO) in Milk of Lime of Different Densities (Mateczek) . . . . .	76
XXII. Table giving the Quantity of Milk of Lime to Add per Hectolitre of Juice Extracted (Unger) . . . . .	77
XXIII. Solubility of Strontia, Strontium Hydrate, and Caustic Baryta in Water . . . . .	83
XXIV. Table giving the Percentage of Lime in Animal Charcoal, working on the Normal Weight of 1.7 Grammes.—For Use in the Special Testing of Animal Charcoal (Scheibler) . . . . .	115

## D. TABLES RELATING TO CONCENTRATION OF BEET JUICE TO SYRUP.

XXV. A = strength of juice or saccharine liquor in solids per cent. B = lb. of water to be evaporated to bring the total solids to 80 per cent. . . . .	125
XXVI. Boiling-points of Pure and Impure Solutions of Sugar . . . . .	128
XXVII. Results of Vaporisation of Water Experiments (Schmidt) . . . . .	130
XXVIII. Concentration of Juice.—A, density of extraction juice. B, hectolitres of juice required to obtain hectolitre of syrup of 25° Baumé, sp. gr. 1.2. C, kilogrammes of steam to evaporate to obtain hectolitre of syrup of 25° Baumé. D, kilogrammes of water to evaporate per hectolitre of juice to convert it into syrup of 25° Baumé. E, kilogrammes of syrup at 25° Baumé obtained per hectolitre of juice . . . . .	131

# LIST OF TABLES

xxv

	PAGE
XXIX. Multiple-effect Evaporation Data (Jelinek) . . . . .	142
XXX. Saving per 100 Kilogrammes of Steam withdrawn from Multiple-effect Plant to Heat other Vessels . . . . .	168
XXXI. Allocation of Gross Steam Consumption to each Depart- ment of a Beet-sugar Factory . . . . .	170
XXXII. Table of Sections to Adopt for the Passage of Steam from the Escape Balloon to No. 1, from No. 1 to No. 2, from No. 2 to No. 3, and from No. 3 to Condenser, for Triple- effects of Different Capacities. The Pressure in the Return Balloon is 500 Gr. Effective. The Vacuum is Nil in No. 1, it is 82.4 in No. 2, and 58.13 c.m. in No. 3 (Cambier) . . . . .	171
E. TABLES RELATING TO EVAPORATION OF BEET SYRUP TO MASSE CUITE.	
XXXIII. Solubility of Sucrose at Different Temperatures, with Den- sities in Degrees Baumé and Degrees Twaddell, and in Actual Gravity at the Temperature of the Experiment, and as brought to 15° C. (Flourens) . . . . .	178
XXXIV. Solubility of Sucrose in Water at Different Temperatures	180
XXXV. Boiling-points of Sugar Solutions in Vacuum Pan at Dif- ferent Pressures of Air . . . . .	180
XXXVI. Composition of <i>Masse Cuite</i> and Syrups of Different Den- sities (Mauméné) . . . . .	197
XXXVII. Density of <i>Masses Cuites</i> and Syrups as Index to Apparent Dry Matter . . . . .	198
XXXVIII. Output of Pumps in Litres per Hour Required to Effect the Returns ( <i>Retours</i> ) from Heating, Multiple-effect, and Strike Pans, and to send them into the Boilers, or, if desired, into the Reservoirs ( <i>Ballon des Retours</i> ) (Cambier) . . . . .	199
XXXIX. Steam Consumed per Ton of Beets in Beet-sugar Factory Working 300 Tons of Beets in 24 Hours.—Kilogram- mes of steam expended: A, in heating and evaporating. B, by engines, partly restored to evaporation. C, restored to evaporation. D, excess of consumption over evaporation. E, actually expended without resti- tution. F, total expended on heating, evaporation and motive power . . . . .	199
XL. Expenditure of Steam by each Engine in a Beet-sugar Works Treating 300 Tons of Beets in 24 Hours.—A, diameter of pistons in mm. B, speed of pistons per second. C, volume generated hourly by pistons in litres. D, volume of steam in litres admitted hourly—admis- sion 80%. E, weight of steam consumed hourly . . . . .	200

	PAGE
XLI. Steam Consumed per Hectolitre of Juice in the Different Branches of a Beet-sugar Works Working 300 Tons in 24 Hours. Juice Obtained per Ton, 1,503 Litres.— Kilogrammes of steam expended: A, in heating triple-effect. B, by engines, partly restored to evaporation. C, kilogrammes of steam restored by engines to evaporation. D, excess of consumption over evaporation. E, kilogrammes of steam actually expended without restitution. F, total steam expended on heating, evaporation and motive power . . . . .	200
XLII. Working Data and Results of Vacuum Strike Pan. A, degree Baumé at exit from multiple-effect. B, quantity of syrup necessary per hectolitre of <i>masse cuite</i> . C, amount of water to evaporate to obtain a hectolitre of <i>masse cuite</i> . D, water evaporated per hectolitre of syrup to transform it into <i>masse cuite</i> . E, <i>masse cuite</i> obtained per hectolitre of syrup . . . . .	201
F. TABLES RELATIVE TO CRYSTALLISATION IN MOTION AND TURBINING OF <i>MASSE CUITE</i> .	
XLIII. Influence of Purity of the <i>Masse Cuite</i> on the Yield in Sugar when the Discharge Syrup has a Constant Purity of 72 . . . . .	217
XLIV. Influence of the Purity of the Discharge on the Yield in Sugar of the Boiled Mass when the Purity of the Latter is Constant . . . . .	217

## SECTION II.

## G. TABLES RELATIVE TO CANE SUGAR.

XLV. Yield of Sugars per Ton of Cane . . . . .	242
XLVI. Résumé Showing the Coal Consumption for each Diffusion Run . . . . .	256
XLVII. Sugar-cane Diffusion—Battery-man's Report Sheet . . . . .	262
XLVIII. Table Showing Comparative Temperature in each Cell in Sugar-cane and Sugar-beet Diffusion Batteries . . . . .	267
XLIX. Résumé Showing the Mean Composition of the Normal and Diffusion Juices, the Maximum and Minimum Density, Percentages of Sucrose, Reducing Sugars (Glucose, etc.), Coefficient of Purity, etc., 75 Analyses (Magnolia, 1888-89) . . . . .	269
L. Evaporation in Diffusion Work as compared with that of 72 Per Cent. Milling . . . . .	270
LI. Magnolia Plantation Crop Report, 1888, Diffusion Runs . . . . .	271

## LIST OF TABLES

xxvii

	PAGE
LII. Available Sucrose (Sucrose minus $1\frac{1}{2}$ times Glucose) . . .	272
LIII. Relation between the Density of the Juice of the Sugar Cane and its Richness in Sugar . . . . .	274
LIV. Showing the Mean Composition of Fourth Cane-Sugar <i>Masses Cuites</i> and Molasses therefrom at Magnolia, 1888-89 . . . . .	281

## SECTION III.

### H. TABLES RELATIVE TO SUGAR REFINING.

LV. Analyses of Raw Sugar . . . . .	284
LVI. Analyses of New Animal Charcoal . . . . .	295
LVII. Analyses of Various Old or Used Bone Chars . . . . .	306

## SECTION IV.

### I. TABLES RELATIVE TO THE CHEMISTRY OF SUGARS, ANALYSES OF COMMERCIAL SUGARS AND OF MERCHANDISE CONTAINING SUGARS.

LVIII. Density of Solutions of Sucrose of Different Strengths at 17°-5 C. . . . .	389
LIX. Corrections of Degrees Balling for Temperatures other than 17°-5 C. (Stammer Sachs) . . . . .	340
LX. Table Exhibiting the Quantity of Sugar, in Pounds Avoirdupois, which is contained in One Gallon of Syrup, at Successive Degrees of Density, at 60° F. . . . .	341
LXI. Solubility of Sucrose in Mixtures of Alcohol and Water . . . . .	343
LXII. (1) Density of Aqueous Solutions of Glucose (Salomon), and (2) Solubility of Glucose at 64° F. in Alcohol of Various Strengths (Anthon) . . . . .	347
LXIII. Graduation and Adjustment of Polariscopes: French and German Scales . . . . .	354
LXIV. Correction to 17°-5 C. of Polarimetric Results for Different Temperatures instead of 100 Pol. Dega. . . . .	360
LXV. Effect of Concentration on Rotary Power of Sucrose . . . . .	361
LXVI. Rotary Power of Sugars, Sucrose, Lactose, Maltose, Glucose, Levulose, etc. . . . .	362
LXVII. Analysis of Commercial Starch Glucose . . . . .	366
LXVIII. Proportion of Cane Sugar contained in Impure Sugars or Saccharine Juices . . . . .	367
LXVIII. Phenomena Observed in Titrating Glucose with Copper Solution (Violette) . . . . .	368

	PAGE
LXIX. Estimation of Glucose in Raw Sugar . . . . .	369
LXX. Volumetric Estimation of Glucose and Levulose, Lactose and Maltose.—(Table for use with solutions containing 0.1 to 0.5 per cent. of reducing sugars—titrated with 10 c.c. Fehling's solution—the results being expressed in grammes per litre or in lb. per 100 gallons.) . . . .	370
LXXI. Gravimetric Estimation of Reducing Sugars . . . . .	376
LXXII. Gravimetric Estimation of Reducing Sugars . . . . .	376
LXXIII. Gravimetric Estimation of Reducing Sugars . . . . .	380
LXXIV. Gravimetric Analysis of Dextrose in Raw Sugar (Allihin) . . . .	381
LXXV. Estimation of Invert Sugar, Calculated from E. Meissl's Data by E. Wein . . . . .	382
LXXVI. Table for Use in Gravimetric Estimation of Maltose . . . . .	384
LXXVII. Table for Use in the Gravimetric Estimation of Milk Sugar . . . .	385
LXXVIII. Gravimetric Estimation of Small Quantities of Glucose (Herzfeld) . . . . .	386
LXXIX. Comparison of Violette's, Fehling's, Knapp's, and Sachs' Solutions . . . . .	387
LXXX. Chemical Composition of Molasses . . . . .	396
LXXXI. Clerget's Table . . . . .	400
LXXXII. Table Syrups—Analytical Data . . . . .	403
LXXXIII. Table Syrups—Composition . . . . .	403
LXXXIV. Analyses of Molasses from Various Sources . . . . .	404
LXXXV. Table for Calculating the Sucrose Corresponding to the Invert Sugar Present from the Copper Found on Three Minutes' Boiling . . . . .	407
LXXXVI. Meissl's Table for Estimating Reducing Sugars in Raw Sugar . . . . .	408
LXXXVII. Determination of Raffinose in Raw Sugar . . . . .	410
LXXXVIII. Allowance for Temperature in Taking Gravity of Syrups . . . .	417
LXXXIX. Table for Use in the Saccharimetrical Examination of Urine.—100 c.c. of Urine Clarified with 10 c.c. of Basic Acetate of Lead . . . . .	424
XC. Analysis of Samples from Different Sources of Fresh and Preserved Bananas . . . . .	425
XCI. Analysis of Commercial Preserved Bananas . . . . .	426

# TECHNOLOGY OF SUGAR.

## SECTION I.

### BEET SUGAR.

#### CHAPTER I.

##### COST OF PRODUCTION—VALUATION—PURCHASE AND PRELIMINARY TREATMENT OF BEETS.

*CULTIVATION of the sugar beet in Britain.*—The first British attempt to manufacture sugar from beets was made by James Duncan, in 1868, at Lavenham, Suffolk. The factory did not survive many years. The beets grown in the neighbourhood yielded fully 10 per cent. of sugar with a crop of from fifteen to eighteen tons per acre. Lawes and Gilbert and the elder Dr. Voelcker long ago proved that the British soil and climate is not unfavourable to sugar-beet culture, and that *beets can be grown in favourable localities in Ireland and even in Scotland as rich in sugar as those of France and Germany.* Why, therefore, continue to endow and repeat experiments to demonstrate acknowledged facts? But British farmers will possibly demur to grow, as at Lavenham, small crops, rich in sugar, if they cannot get more than twenty shillings per ton delivered, a seemingly good price, and a good profit, but cartage may average five shillings per ton, leaving only fifteen shillings per ton clear. If it suit Continental farmers to sell beets at fifteen shillings a ton it does not do so here, where it pays better to convert beets into beef. Again, where sufficient cattle food cannot be raised for fattening purposes and big sums are spent in buying oil-cake, etc., a farmer cannot be expected to sell beets at twenty shillings per ton delivered unless like the Continental farmer he be a shareholder in the sugar factory (p. 6) and recoup himself for his loss on beets by his sugar-factory dividends. When this is realised there will be a grand future in Britain both for sugar-beet growing and beet-sugar manufacture, provided always they be conducted on rational principles and with unstinted capital. Beets can be grown to perfection even in Canada, but the maltreatment they received through inexperienced immigrant labourers led, it is said, to the closing of at least one factory there.

*Cost of growing beets: (1) In Europe.—(a) In France,* according to Malpeaux, it is rather a difficult matter to ascertain exactly the outlay entailed and the profits realised by the cultivation of the sugar beet. The factors on which the calculations are based are so variable that any generalisations in regard to these points are very risky. Any such estimate must be heavily discounted, because it is impossible to debit any one crop with its exact share of the expenses. Moreover, the horses and permanent staff of the farm would often be standing idle during the beet cultivation season were it not for such operations as horse-hoeing, drill harrowing, etc.

One thing is certain, and that is, that the outlay is always very heavy, and is almost sure to vary with the locality. There is the difference in the rent of land to be considered, the cost of labour, and the method of cultivation adopted. The chief reasons which cause the heavy outlay to be incurred are the large quantities of artificial manure that have to be used, and the many hoeings, etc., etc., which the growth of the beet entails. Again, it must be borne in mind that the beet only grows to perfection on deep soils of excellent quality, thus entailing a heavy rental. It is therefore necessary to secure a heavy crop of roots, rich in sugar, as they are sold according to the sugar content which, if in direct ratio to the density of the root, is unfortunately in inverse ratio to the weight of the crop.

As matters stand at the present time, good farmers can hardly hope to get a crop exceeding 30,000 kilogrammes per hectare (say about 12 tons per acre), the varieties cultivated since 1884 being much less productive than those in vogue prior to that date. The average weight of the crops for the whole of France was 25,142 kilogrammes per hectare (say 10 tons per acre) for 1899, but it varies with the season and the weather. Thus in 1889 it reached the figure of 31,565 kilogrammes (say 12½ tons per acre), whilst in 1893 it fell to 23,300 (say 9 tons per acre).

The yield not only varies with the weather, but also with the locality, in the same year. This is due to the nature of the soil, to the varieties grown and the method of cultivation. In this connection there is a decided difference between Departments. Thus in 1898 the yield was 30,000 kilogrammes per hectare (say about 12 tons per acre) in the Nord and the Oise, whilst it was 28,000 in the Eure and Calvados, and 26,000 in the Pas de Calais, Seine et Marne, and Seine et Oise, 23,000 in the Somme, and 22,000 in the Aisne. The average yields, therefore, bear no ratio to the amount of land under beet.

The average weight of the crop per hectare of land in good cultivation cannot be estimated at more than 28,000 to 30,000 kilogrammes (say 11 to 12 tons per acre), which, in taking 25 francs per 1,000 kilogrammes at 7° (say £1 per ton) as the price basis, gives a

gross return of 700 to 750 francs per hectare (say roughly £11 to £12 per acre).

If, continues Malpeaux, we place against these figures the prime cost of a hectare of beets, we find, from the figures laid before the Agricultural Committee of Laon in 1895, we get the following:—

TABLE I.—*Cost of growing a hectare of sugar beets in the Plain of Laon, France.*<sup>1</sup>

(A, francs per hectare; B, shillings and pence per acre.)

	A.	B.
		s. d.
Rents and taxes . . . . .	80	25 6
Measuring . . . . .	1	0 4
Supervision . . . . .	4	1 3
Farmyard dung and covered in manure . . . . .	116.66	37 3
Surface manure . . . . .	177.50	57 0
Sowing, including seed and labour . . . . .	45	14 4
Hoeing and lifting . . . . .	95	30 5
Horse hiring . . . . .	25	8 0
Cartage . . . . .	75	24 0
Sundry expenses . . . . .	123.34	39 6
	<hr/>	<hr/>
	742.50	237 7
Deduct 20,000 kilogrammes of leaves at 3 francs		
the 1,000 kilogrammes . . . . .	60	19 3
	<hr/>	<hr/>
	682.50	218 4

Taking everything into account, therefore, the cultivation of the sugar beet lately would not be very profitable were it not that exceptional climatic conditions enabled the farmers to obtain roots of greater density than 7<sup>3</sup> (see p. 10), and thus to profit by the enhanced price given by the manufacturers.

(b) *In Germany* the culture of the sugar beet is generally more lucrative than in France, looking to the price given by manufacturers, the lower price paid for labour, rent and taxes, and the heavier crops got per acre. Notwithstanding the magnificent results obtained in France since the inauguration of the new régime in 1884, the French farmer still lifts a crop which weighs 3,809 kilogrammes per hectare (say 1½ tons per acre), or 14.3 per cent. less than that of the German farmer, and delivers to the sugar manufacturers beets of which 160 lb. more are required than in Germany to produce 100 lb. of sugar.

(2) *In U.S.A.*, Wiley gives the following estimate of the maximum cost per acre, based on the supposition that land is worth \$75 (£15) per acre, labour \$1 (say 4s.) per day, the yield 15 tons per acre, and that the distance to the factory is not greater than three miles:—

<sup>1</sup> A hectare is roughly 2½ acres (2.47); 1,000 kilogrammes is 2,200 lbs., i.e., a metric ton, and 25 francs = £1. The dollar is taken as 4s.



## TECHNOLOGY OF SUGAR

TABLE II.—*Cost of growing sugar beets in the United States.*

(A, in U.S.A. dollars; B, in shillings per acre.)

	A. Per acre. \$	B. Per acre s.
Rent of land . . . . .	5-00	20
Ploughing, subsoiling and preparing for planting . . . . .	5-00	20
Fertilisers . . . . .	10-00	40
Cost of seed . . . . .	2-25	9
Planting . . . . .	1-75	7
Hoeing and thinning . . . . .	6-00	24
Cultivating with horse-hoe five times . . . . .	5-00	20
Harvesting . . . . .	5-00	20
Topping . . . . .	12-00	48
Delivery to factory . . . . .	7-50	30
<b>Total . . . . .</b>	<b>\$59-50</b>	<b>238</b>
Value of 15 tons at \$4-50 . . . . .	67-50	270

In the above estimate the cost of the beet culture and a reasonable application of fertilisers has been included. Wiley thinks that the actual cost to American farmers for the first few years of the beet industry will not exceed \$45 or \$50 (say £9 to £10) per acre. The price paid in America for the beet is dependent on many local considerations, but chiefly on the selling price of sugar and the richness of the beet. It is reasonably certain, continues this authority, that a net profit of from \$8 to \$15 per acre may be expected from the proper culture of the sugar beet in localities near a factory when all the conditions of the best methods of culture are fulfilled.

Wiley goes on to quote and contrast with the above estimate the actual figures submitted by J. Thomson of Hall County, Nebraska, in a letter published in the *Prairie Farmer* on the 3rd January, 1891. The cost is given for a field of 5 acres as follows :—

TABLE III.—*Actual cost of growing sugar beets in U.S.A.*

(A, in U.S.A. dollars; B, in shillings per 5 acres.)

	A. \$	B. s.
Fall ploughing at \$1 per acre . . . . .	5-00	20
Ploughing in spring, \$1-25 . . . . .	6-25	25
Rolling twice, once before and once after planting . . . . .	1-50	6
Planting by hand at 75 cents per acre . . . . .	3-75	15
Cultivating with hoe at 75 cents . . . . .	8-75	15
Thinning at \$10 . . . . .	50-00	200
Hoeing by hand three times and weeding necessary at time of hoeing . . . . .	90-00	360
Cultivating by horse twice at 75 cents . . . . .	7-50	30
Running over with hoe to clear remaining weeds . . . . .	8-75	15
	<b>\$171-50</b>	<b>686</b>

Dividing this by 5 gives a total of \$34-30 (137s. 3d.) per acre.

The criticism states that Thomson makes no allowance for the rent of land used, no fertilisers, and gives no estimate of the price of seed

# COST OF GROWING SUGAR BEETS

5

and of the expense of harvesting and hauling to the factory. But this field was hoed by hand three times, which, with the proper implements, is more than is necessary, one good hand-hoeing being as a rule sufficient.

Wiley quotes the following estimate by a French statistician:—

TABLE IV.—*Cost of growing sugar beets—a French estimate.*  
(A, in U.S.A. dollars; B, in shillings and pence per acre.)

	A.	B.
	\$	s. d.
Farmyard manure . . . . .	28-00	112 0
Commercial fertilisers . . . . .	12-00	48 0
Spreading manure . . . . .	0-60	2 5
Spreading fertilisers . . . . .	0-25	1 0
First ploughing . . . . .	1-60	6 5
Harrowing and rolling after ploughing . . . . .	0-64	2 7
Ploughing and subsoiling . . . . .	4-60	18 5
Two scarifyings . . . . .	2-00	8 0
Two harrowings . . . . .	0-64	2 7
Two rollings . . . . .	0-64	2 7
Cost of seed . . . . .	3-00	12 0
Sowing of seed . . . . .	0-80	3 2
Harrowing and rolling again . . . . .	0-64	2 7
Three times horse-hoeing . . . . .	2-40	9 7
Hoeing by hand . . . . .	4-80	19 2
Harvesting by machine . . . . .	1-60	6 5
Lifting and topping . . . . .	4-00	16 0
Cartage to factory . . . . .	2-40	9 7
Rent and taxes . . . . .	9-40	37 7
	<hr/> \$80-01	<hr/> 920 1

The charge for harvesting is very low, as the topping of the beets is performed by women and children, whose average wage probably does not exceed 15 cents a day. As, however, women and children respectively earn about double this in the sugar factories, they can surely hardly be got to work in the fields for half that amount. Wiley is therefore probably in error in saying that women at any rate can be got to work in the French fields for  $7\frac{1}{2}$ d. per day. Be that as it may, the charge for manures is, as Wiley states, much greater than would be necessary in America for some years to come. The average yield per acre obtained by Du Fay, the author of the above estimate, was 25 tons per acre,  $62\frac{1}{2}$  tons per hectare, thus illustrating the value of intensive farming. But Wiley does not give the sugar content, and  $62\frac{1}{2}$  tons per hectare is incompatible with the growth of rich beets.

*Return on capital invested.*—The cost of manufacture in U.S.A., says Wiley, depends on as many factors as that of beet-growing, chiefly transportation, fuel, weather and labour. Perhaps the most important is the price of fuel. In some localities coal can be had for \$1-25, say 5s., per ton; in others the cost may reach as high as \$10, say £2, per ton. Beet sugar is manufactured without government supervision in the U.S.A., and its exact cost is inaccessible.

With experience, highest skill and management, the mean cost of manufacture in 113 German factories is given as:—

TABLE IVa.—*Return on Capital Invested.*

	£	s.	d.	\$
Mean capital invested in each factory . . . . .	40,291	13	4	139,400-00
Total receipts for sugar, molasses, etc., per ton of beets . . . . .	2	6	3	11-10
Mean cost of beets per ton of 2204-62 pounds . . . . .	1	0	5	4-90
Salaries per ton of beets . . . . .	0	1	1	26
Labour per ton of beets . . . . .	0	3	0½	73
Interest on investment per ton of beets . . . . .	0	1	6	36
Coal per ton of beets . . . . .	0	2	7½	63
Miscellaneous expenses per ton of beets . . . . .	0	4	0	96
Total expense of manufacture per ton . . . . .	1	12	8	7-84
Profit per ton of beets . . . . .	0	13	7	3-26

The mean nett profit for each factory was \$94,240 (£7,133 6s. 8d., or 17½ per cent. on invested capital). The price paid for beets is often fictitious, the beet-growers owning the factory, and preferring to share in its profits rather than charge high for the beets. First-class beets rarely sell for less than \$5 (£1) per ton. The Western Beet Sugar Company, of Watsonville, Cal., stated that in its first campaign, 1888-89, the cost of manufacture amounted to \$80-80 (£16 16s. 8d.) per ton of sugar. At the present time it appears that, with the best machinery and most economical processes, beet sugar can be made in U.S.A. at a cost of from 3 to 4 cents (1½d. to 2d.) per pound when the price of rich beets does not exceed \$5 (£1) a ton.

*Cost of factory.*—The cost of building a first-class beet-sugar factory is much greater than is commonly supposed. From the most reliable data, in Europe, the cost of erecting a factory with the most modern machinery, of a capacity of at least 300 tons of beets per day, is about \$200,000 (£40,000). In U.S.A., owing to the increased cost of transportation and the higher price of labour, the cost of a similar factory would be at least \$250,000 (£50,000). It is not advisable to attempt to manufacture beet sugar with smaller factories or with machinery and appliances which do not represent the latest improvements. If many parts of a sugar factory have not changed much within the last twenty years, even the multiple-effect apparatus, the strike pans and the centrifugals, which represent the most stable parts of the machinery, have undergone considerable changes within that time. Probably one of the greatest dangers which the beet-growing industry in the U.S.A. will meet is the tendency to begin the erection of a beet-sugar factory with cheap, old or worn-out appliances, and without a proper technical study of all the questions involved. This is the

more difficult to avoid because there are few engineers in the U.S.A. who have devoted themselves to the study of this problem, and European experts are not likely to understand and comprehend American methods and measures. Numerous inquiries are made for directions for making beet sugar with such appliances as a cider mill and sorghum-molasses evaporator. It would not be right to encourage the attempt to manufacture beet sugar in any such way. Nor should the expectation be excited among farmers that they will be able to make a raw sugar which they can dispose of to a central factory for refining purposes. It is best to recognise at once the great expense attending the erection of a sugar factory and the necessity for its meeting every modern requirement. Beet-growing and beet-sugar manufacture are two distinct industries, but with common aims and interests.

#### VALUATION AND PURCHASE OF SUGAR BEETS.

*Purchase of beets by contract.*—*The contract specifications.*—Sugar beets are sold by contract. The farmer agrees to grow sugar beets under predetermined conditions, at a price fixed beforehand. At the time of laying down the crop the manufacturer, on the one hand, endeavours to make himself sure of getting prompt delivery of a certain quantity of beets, so as to enable him to negotiate, on a certain quantity of sugar, to be delivered after manufacture. The farmer, on the other hand, seeks an outlet for his crop. They enter into a mutual agreement by which the farmer, on his part, engages to deliver, beginning with a stated period, sugar beets from such and such a field of a determined composition, at the price previously stipulated. The farmer is generally free to till the ground as he likes, and to use whatever manure suits him best. The manufacturer, on his part, supplies the seeds, so as to ensure the authenticity and sugar content of the varieties cultivated.

*The farmer's risk in waiting to secure the current market price.*—Some farmers do not enter into an agreement. They prefer to wait until the September sales, so as to discuss the price of their beets by the prices ruling in the sugar market. But they may be at the mercy of the manufacturer, if they do not wish to endanger their crop. It is therefore prudent for them to sign an agreement.

*Delivery.*—The roots are often carted to the factory, but, unless the fields be quite near, that is a great expense which certain factories try to reduce by establishing depôts at their own cost, provided with steelyards, near to the farms and on the most frequented highways. Sometimes the beets are brought by rail or boat. The methods of transporting the beets from the depôts to the factory will be discussed later on.

*Sampling.*—The deliveries are sampled in the cart, in the railway waggon, in the field, or in the pit or silo.

*Sampling in the cart or railway waggon.*—When beets are sold in the railway waggon or cart, a determination of the density or the richness in sugar is made on each waggon, or every two or three carts, choosing the roots from the whole bulk, so as to obtain a fair average sample.

*In the field.*—Sampling is sometimes done on the field. A certain number of roots are pulled, arranged in lines or rows, in order of size; then the tenth, the twentieth, etc., taken, so as to get 25 to 30 roots per hectare (say 10 to 12 per acre). The sampling may be done similarly before pulling the roots, by starting in a direction chosen at random, and proceeding in a straight line.

*Sampling the deliveries at the factory.*—The manufacturer always reserves the right of controlling the density, as per sale contract, by taking samples at the time of delivery of the roots to the factory.

*Tare (allowance to be deducted for mud).*—A certain amount of soil always adheres to the beets when pulled, more especially after rain, when they sometimes bring with them a considerable weight of soil. The weight of this mud must be deducted. To ascertain exactly the weight of roots available for manufacture, a certain weighed quantity is taken on delivery, say a basketful of beets from all over a cart. These beets are weighed, washed in a drum with rotating blades, then scraped, brushed and reweighed, and the difference gives the tare, i.e., the mud which the beet has brought along with it.

*Heading and tailing the beets at the factory.*—Before the second weighing the beet is stripped of any remaining leaves, and, unless the operation has been previously satisfactorily done on the field, the beets are "headed" again, i.e., the top of the beet is cut off flat, perpendicular to the axis at the base of the first leaf. This should be done on the field, when pulling the beets. The top conical part of the beet, bearing the leaves, contains but little sugar, and is, moreover, highly charged with injurious salts. Therefore, if the heading has not been satisfactorily done on the field, this important operation is completed at the factory. The beets thus cleaned, trimmed and weighed to get at the tare, are used for the analysis, when the beets are bought according to their sugar content. By a recent legislative enactment, all steelyards used in weighing beets in France must be furnished with a registering apparatus, working so as to successively print, on the same ticket and on lines sufficiently far apart, the weight of the cart when full and when empty.

These operations, relative to the weighing, the waste and the density, ought, in France, to take place under the control of the excise-man attached to the factory. The manufacturers are bound to facilitate this control and to provide the necessary instruments. But by a tacit agreement between farmers and manufacturers there are but few factories which conform to these arrangements for the reception of beets by the excise officials. The excise arrangements

for weighing the beets as they pass in for treatment are detailed on p. 30.

*Sale of beets by analysis.*—The basis of the sale of beets depends on their sugar content. To determine this, three methods may be adopted: 1. The determination of the density of the juice; 2. The saccharimetric value of the juice; 3. The direct analysis of the pulp.

1. *Estimation of the percentage of sugar in beets from the density of the expressed juice.*—Beets are purchased by the density of the juice in France since the legislative enactment of 1884 came into force, although it is a well-known fact that there is no well-determined ratio between (1) the density of the juice, and (2) the actual percentage of sugar in the beet. This ratio may, in fact, vary from 1.75 to 1.97 per unit of density, the sugar being, of course, calculated on the weight of the root. When it is a case of determining the sugar content of a beet, so as to decide whether it should be selected as a seed producer, polarimetric methods (Fig. 4) must be adopted.

TABLE V.—CORRECTIONS TO BRING DENSITY OF BEET JUICE AT VARIOUS TEMPERATURES TO DENSITY AT 15° C.

° C.	Correction.	° C.	Correction.	° C.	Correction.	° C.	Correction.
	Minus.		Minus.		Plus.		Plus.
0	0.20	11	0.09	21	0.15	31	0.43
1	0.19	12	0.07	22	0.17	32	0.46
2	0.18	13	0.05	23	0.20	33	0.49
3	0.17	14	0.02	24	0.22	34	0.52
4	0.16	15	0.00	25	0.25	35	0.55
5	0.15		Plus.	26	0.28	36	0.60
6	0.14	16	0.02	27	0.31	37	0.64
7	0.13	17	0.05	28	0.34	38	0.67
8	0.12	18	0.07	29	0.37	39	0.70
9	0.11	19	0.10	30	0.40	40	0.74
10	0.10	20	0.12				

*Influence of temperature on density.*—The density is taken by a hydrometer, which marks 0° in distilled water at 15° C. At other temperatures a correction is necessary, because this liquid expands and becomes lighter as the temperature rises, and *vice versa*. Tables have been constructed giving the corrections to be made, but they cannot be quite exact, as the expansion of the juice varies with its composition. It has been suggested that it would be sufficient to add or deduct 0.1 from the gravity found, according as the temperature is 4° above or below 15° C. The use of the above table, however, has been made obligatory in the case of beet deliveries in France by legislative enactment, promulgated on the 23rd July, 1897.

*Explanation of table.*—Suppose the hydrometer shows a density of 1.075, which is called 7.5 excise degrees. Let the temperature be 19° C., 19 is found in the temperature column, and in the other column it will be seen that it is necessary to add 0.1 excise degrees to the result found, which gives for the density of the juice 7.5 + 0.1 = 7.6 excise degrees, or an actual density of 1.076. But suppose, on the other hand, that the density found is 1.085, which is announced as 8.5 at the temperature of 9° C., the corrected density will be 8.5 - 0.11 = 8.39 excise degrees, or an actual density of 1.0839.

According to contracts, if it be a case of purchase the density will be taken at 8.3 or 8.4 instead of 8.39. The determination of the density of the juice, although apparently a simple operation, requires certain precautions to secure accuracy.

*Preparing the samples of beets.*—The first thing is to well clean and head the beets which form the lot to be analysed, and which should not be less than five. If the lot is not a big one the whole of the roots are pulped. Otherwise only half or quarter of the roots, cut exactly in the direction of their length, are pulped, placed in a stout calico bag, and strongly pressed in a screw press with a spout through which the juice flows into a collecting vessel. The delivery is then paid for according to their density, an average rate as a basis of price, for example, being 25 francs (say £1) per metric ton of beets of 6° (that is, sp. gr. 1.06), and reducing or increasing this price for each tenth of a degree greater or less.

*The first and final flow differ in density.*—From Saillart's experiments at the French National School of Agricultural Industry, it appears that the first flow of juice is always of greater density and is richer in sugar than the final flow. The variation may be as much as  $\frac{1}{10}$ . The juice is run into a tall cylindrical jar, wide enough for a hydrometer to float in it easily, filling the jar to the brim, so that the liquid flows over. The froth on the surface is blown off and the operator waits ten to twelve minutes before inserting the hydrometer, so that no more froth (due to air-bells suspended in the liquid, which might reduce the density by several tenths of a degree) may be formed.

*The correct method of reading the hydrometer meniscus.*—When the hydrometer is immersed in the liquid, the latter rises against the sides of the jar, forming the *meniscus*. The reading should not be taken from the top of the attenuated layer around the stem, but on the prolongation of the horizontal surface of the liquid, always easy to discern, with sufficient approximation, in graduated hydrometers.

*A prevalent fallacy and its effects.*—Certain farmers wrongly imagine that the percentage richness in sugar of the beet may be got by multiplying the density by 2. A custom has thus arisen in France whereby the percentage of sugar divided by 2 is taken as the density. The error arose from a loose habit of some chemists of inserting in their reports of the indirect analyses of beet the per-

# SACCHARIMETRICAL VALUATION OF BEETS 11

centage of sugar in c.c. of juice—a figure of no great value—and of dividing this result by the density. Coefficients were thus got varying from 2 to 2.2, which amounts to saying that in 100 c.c. of juice extracted from the beets in question there was a quantity of sugar equal to 2 to 2.2 times the density.

*The percentage of sugar in juice of given density not always uniform.*—The same density is far from corresponding to the same percentage of sugar, and the error may amount to two units. The sugar content per cent. of the beet is, on an average, inferior or superior to the degree multiplied by 2, as that degree is lower or higher than 7.5. The density alone is not sufficient to give an exact idea of the composition of the sugar beet. However good for giving the farmer some information as to the quality of his beets, it is an uncertain basis for contracts.

*Simplicity its chief recommendation.*—An argument, however, in favour of this method is its simplicity. It requires no laboratory apparatus, and it has the advantage of both contracting parties being able to follow and understand its operations, which can be executed and discussed under the eyes of those concerned.

2. *Saccharimetrical valuation.*—Sometimes the juice is tested optically (Fig. 4, p. 19), and the farmer is paid on the polarisation results. But this is exceptional. The farmer will have nothing to do with the results of an instrument the use of which he in no way understands, and in which he sees nothing but “looking through a lens at a wafer cut in two”.

TABLE VI.—ESTIMATION OF THE AMOUNT OF SUGAR IN BEETROOTS (EDOUARD DELVILLE).

Density.	Factor.	Density.	Factor.	Density.	Factor.
1.04	0.164	1.057	0.162	1.074	0.159
1.041	0.164	1.058	0.162	1.075	0.159
1.042	0.164	1.059	0.161	1.076	0.158
1.043	0.164	1.06	0.161	1.077	0.158
1.044	0.163	1.061	0.161	1.078	0.158
1.045	0.163	1.062	0.161	1.079	0.158
1.046	0.163	1.063	0.160	1.08	0.158
1.047	0.163	1.064	0.160	1.081	0.158
1.048	0.163	1.065	0.160	1.082	0.158
1.049	0.163	1.066	0.160	1.083	0.157
1.05	0.163	1.067	0.160	1.084	0.157
1.051	0.162	1.068	0.159	1.085	0.157
1.052	0.162	1.069	0.159	1.086	0.157
1.053	0.162	1.07	0.159	1.087	0.157
1.054	0.162	1.071	0.159	1.088	0.157
1.055	0.162	1.072	0.159	1.089	0.156
1.056	0.162	1.073	0.159		



This table (VI.) is for use with saccharimeters of the Duboscq-Soleil type, with a normal weight of 16.35 grammes, and supposing the beetroot to contain 0.95 of its weight of juice, the figure on which transactions are generally based. To calculate the percentage of sugar by means of this table, two determinations are required: (1) The density of the juice at 15° C.; (2) the polarisation of the juice after adding  $\frac{1}{10}$  of basic acetate of lead. These having been made, the density is sought in the table, and the number of degrees on the scale of the saccharimeter is multiplied by the factor opposite the density. The product gives the percentage of sugar in the beet.

*Example.*—The juice marks 1°05 density. The saccharimeter shows 60°. The percentage of sugar in the beet is  $0.163 \times 60 = 9.78$  per cent.

TABLE VII.—DENSITY OF SOLUTIONS OF SUCROSE IN WATER AT 15° C.  
(SCHEIBLER).

Per Cent.	Density.	Per Cent.	Density.	Per Cent.	Density.	Per Cent.	Density.
0	1.00000	19	1.07907	38	1.16960	57	1.27297
1	1.00890	20	1.08854	39	1.17470	58	1.27879
2	1.00783	21	1.08804	40	1.17985	59	1.28465
3	1.01178	22	1.09257	41	1.18503	60	1.29056
4	1.01576	23	1.09713	42	1.19024	61	1.29650
5	1.01978	24	1.10173	43	1.19550	62	1.30248
6	1.02382	25	1.10635	44	1.20079	63	1.30850
7	1.02789	26	1.11101	45	1.20611	64	1.31457
8	1.03199	27	1.11571	46	1.21147	65	1.32067
9	1.03611	28	1.12044	47	1.21687	66	1.32682
10	1.04027	29	1.12520	48	1.22231	67	1.33301
11	1.04446	30	1.12999	49	1.22779	68	1.33923
12	1.04868	31	1.13482	50	1.23330	69	1.34550
13	1.05293	32	1.13969	51	1.23885	70	1.35182
14	1.05721	33	1.14458	52	1.24444	71	1.35817
15	1.06152	34	1.14952	53	1.25007	72	1.36457
16	1.06586	35	1.15448	54	1.25574	73	1.37101
17	1.07023	36	1.15949	55	1.26144	74	1.37749
18	1.07464	37	1.16452	56	1.26718	75	1.38401

*The analysis of the expressed juice or the indirect analysis of beet.*—The percentage of sugar in juice may be determined very accurately by the saccharimeter,—100 c.c. of juice used for the density determination are run into a flask, graduated in two places, one at 100 c.c. and the other at 110 c.c. The solution is made up to 110 c.c. by addition of 10 c.c. of basic acetate of lead of 28° or 30° Baumé. The flask is agitated and left to stand until some colourless points are perceived in the greenish mass. It is then filtered, and the filtrate polarised in a 200-millimetre tube. If the solution is cloudy

it is clarified with a drop or two of acetic acid. The sugar per 100 c.c. of juice is found by increasing the result, by  $\frac{1}{10}$ , to make allowance for the basic acetate of lead (see Table XX.).

The sugar content of the juice does not represent that of the beet, and it is the latter that the manufacturer wants to know. The amount of juice in the root is taken as somewhere about 95 per cent. of its weight, and the juice extracted from the pulp by pressure is taken as similar enough to that left behind. But two causes of error may influence the result: (1) The proportion of juice is not always exactly 95 per cent., and (2) the juice obtained by pressure is richer in beginning of flow than at end, as pointed out in 1896 by Pagnoul. Again, the juice, whilst it becomes less rich, also becomes more pure; hence the density decreases with greater speed than the richness. There is thus no homogeneity between the extracted juice and the total juice contained in the root. To deduce the percentage of sugar in the beet from that in the juice, it is necessary to use a coefficient which represents the proportion of juice in 100 of pulped beet, to find which the percentage of sugar in the juice is determined, then, by direct analysis, that in the pulp, and by the formula already given, the number by which it is necessary to multiply the first to get the second. In 82 comparative analyses on pulp and juice Pagnoul found this coefficient in 78 cases included between 91 and 95, and grouped round 93. In no case did he find a lower figure than 90. In indirect analysis he suggests the coefficient 93, not as representing the quantity of juice per cent. contained in the beet, but to pass from the richness of the juice to that of the beet.

*Influence of the precipitate formed by acetate of lead upon polarisation.*—Let S equal the weight of 100 c.c. of juice; B, the weight of 10 c.c. acetate of lead solution; N, the weight of the precipitate formed; F, the weight of the filtered liquid; then  $S + B = N + F$ . If for F we substitute the product of the volume V by the density  $d$ , we obtain  $S + B = N + Vd$ . If the volume of the precipitate be represented by  $v$ , we have the equation  $V + v = 110$ , whence  $V = 110 - v$ . From these equations the following one is derived—

$$v = \frac{110d + N - (B + S)}{d}.$$

From this formula the volume of the lead precipitate is easily deduced.

*Example.*—Weight of 100 c.c. of juice plus 10 c.c. of lead acetate = 118.509 grammes =  $S + B$ . Density of filtered liquid = 1.061 =  $d$ . The weight of the dried precipitate was found to be 3.005 =  $N$ , then by the formula above  $N = 1.136$ . The lead precipitate thus occupies a little more than 1 per cent. of the total volume.

*Purity, non-saccharine, quotient of purity.*—From the density and

the percentage of sugar in the juice the *purity* may be deduced, *i.e.*, the relation between the *weight p* of the sugar contained in 100 c.c. of the juice, and the *weight P* which 100 c.c. of a solution of pure sugar should contain in order to have the same density. The purity will there

fore be expressed by the equation  $\frac{p}{P}$ , and the difference,  $P - p$ , will

be what is called *non-saccharine*. The non-saccharine is therefore got by deducting the polarisation sugar from the figures representing the dissolved matter, both sugar and non-saccharine being calculated on the same basis of 100 c.c. or 100 grammes. The *quotient of purity* represents the percentage of sugar in the dissolved matter. It is ascertained by the formula—

$$Q = \frac{100 S}{\text{Dissolved matter}}$$

To get the weight *P*, consult the tables which give for each degree of density the weight of sugar to make 100 c.c. of the solution. The figures contained in these tables may be approximately obtained by multiplying the French Excise degree by the coefficient 2.6. Pagnoul (see p. 10).

3. *The direct analysis of the sugar beet.*—The difficulties in obtaining invariable results by operating on the pressed juice has already been mentioned (p. 13). When beet pulp is pressed under great pressure the juice which exudes at the beginning is not the same as that which exudes at the finish: the final juice is not so dense as the initial. If the pulp be pressed (*a*) by hand, (*b*) under an ordinary hand press, (*c*) under a powerful hydraulic press, each of the three methods will yield a different liquid. But if it be customary to use a hand press, always turned by the same individual, who as a rule always exerts the same pressure, then the results are comparative, so far as that factory is concerned, although they are not so with those of a neighbouring factory using a hydraulic press.

*Preparation of pulp for analysis.*—In factory pulping of beets for analysis Pellet's pulper (Fig. 1) is used. In its present general form it was in use prior to his aqueous digestion process (p. 17). Primarily intended for the pulping of beets, the juice of which was to be extracted by pressure, it was designed to get a slice throughout the whole of the length of any given beet from the centre to the circumference, and radiating under a given fixed angle representing about  $\frac{1}{3}$  of the beet to be analysed. The cutting is therefore done at an angle of about 30° throughout the whole length of the beet. He made a toothed disc, thick in the centre, diminishing to zero at the circumference. In turning it, if a beet were passed in from the head to the tail, and going as far as the centre, a slice would be cut off over the whole of its half surface of the same shape and

the same relation to the beet as that made by any other beet presented to the instrument in the same way, Pellet's disc, erected like a circular saw for woodwork, is used in factories when there is a large quantity of beets to pulp for analysis.

*Analysis of beets intended for beet seed-producers.*—In the analysis of beets selected for seed-producers a pulp drill fitted up in the

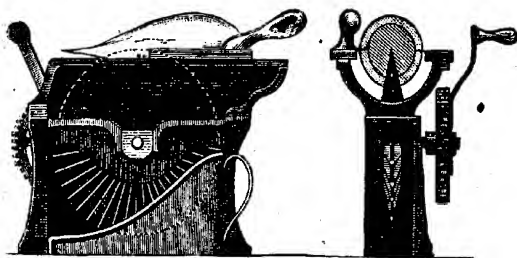


FIG. 1.—Pellet's beet pulper for preparing samples of beet for analysis.

same way is used, only, as the pulp thus formed has to be collected in the hole made by the drill in the beet, the revolving rod of the drill and the drill itself are hollow so that the rasped pulp traverses the drill and is lodged in the hollow rod. When the hole is pierced the rod is withdrawn, the pulp in the interior extracted, and the sample for analysis is obtained.

*Hauriot's apparatus.*—Fig. 3 consists of a truncated steel cone

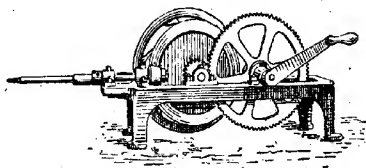


FIG. 2.—Mechanical drill for sampling beets.

Keil-dressed, turning vertically, against which the small pieces of beet taken out by the drill from the mother-root are pressed. The truncated cone is adjusted to a frame carrying the driving gear A, the pusher and a caoutchouc pear P for the wash water, the whole within a small compass so as to facilitate operations. The cylindrical slice obtained by drill shown in Fig. 2 is weighed, and an exact

length giving desired weight is measured and introduced into the pulper, and its pulp washed into the 50 c.c. flask. The apparatus is then washed with water, the washings falling into the flask so that these preliminary operations are done in the twinkling of an eye.

*Pellet's adaptation.*—To avoid weighing the cylinder *Pellet* uses a knife with two parallel adjustable blades. He bases this process on the fact that the difference in weight between two small

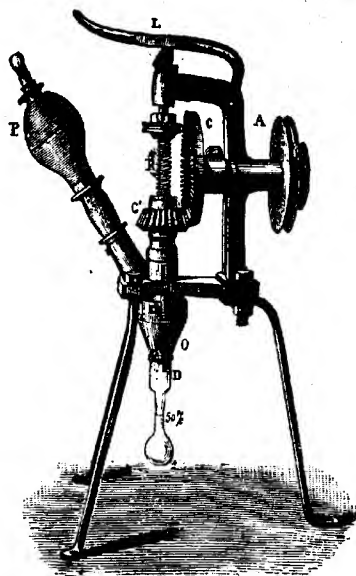


FIG. 3.—Hanriot's apparatus for pulping beet samples.

cylinders of beet of the same diameter and of the same determined length is unappreciable. Hanriot's apparatus (Fig. 3) can pulp 2,200 and even 3,000 cylinders a day. A knife can cut 15,000 to 18,000 cylinders per ten hours' day.

The juice should be collected in a 200 c.c. flask, instead of 100, so as to diminish the strength of the liquid by one-half. Frothing is thus prevented, but the result must be doubled. It is preferable to use a saccharimetrical tube of double the length, so as to be more

## TESTING BEET SEED PRODUCERS

exact and avoid calculation. The direct analysis of the pulped beet by Pellet's aqueous digestion method is to be preferred to the analysis of the expressed juice.

*Pellet's hot aqueous digestion process.*—Principle on which it is based.

—This process is based on the fact that all the sugar in the more or less finely divided pulp is entirely soluble in hot water of 70° to 80° C., the digestion being continued for a quarter of an hour to one hour. The extract is cooled and made up to a given volume, taking into account not only the volume occupied by the insoluble marc, but also that occupied by the precipitate produced by the basic acetate of lead added before digestion, so as to precipitate a large proportion of the extraneous matter, which possesses a polarising power of 3.75, sugar being taken as unity. The hot aqueous method is very exact, but not quite simple enough for certain purposes. When a cream pulp is to be tested, the cold digestion process is sufficient to dissolve all the sugar.

*Pellet's hot diffusion process.*—A certain weight of the pulp produced by a Pellet and Lomont's pulper (Fig. 1), and made homogeneous by stirring, is weighed out, say 16.29 grammes or 32.58 grammes for Laurent's saccharimeter. The pulp is then transferred to a 100 or 200 c.c. flask, with 5 to 6 c.c. of a solution of basic acetate of lead of 28° to 30° Baumé.  $D = 1.241 - 1.262$ . It is made up almost to the mark with water, and kept in a water bath at a temperature of 80° C. for 30 to 45 minutes. The cooled liquid, to which a drop of ether has been added to disperse the froth, is made up with water to 100.9 or 200.7 c.c., so as to take the insoluble into account. Sidersky allows 200.8 c.c. for 16.29 grammes, and 201.35 c.c. for 26.048 grammes, and if the flask be not graduated to these marks, he takes 16.19 instead of 16.29 grammes, and 25.87 instead of 26.048 grammes. The flask is shaken, then its contents are passed through a filter; after throwing away the first drops, which are generally turbid, a clear liquid is obtained, with which the 20 c.c. polarimeter tube is filled. The reading of the saccharimeter gives directly the percentage of sugar.

*Pellet's instantaneous cold diffusion process.*—This simple and correct method—the most practical of all—is now very generally adopted. It gives results equal, if not superior, in accuracy to the most intricate of previous methods. All the sugar in sugar-beet pulp of a certain degree of fineness, such as is obtained by passing it over a somewhat blunt saw, is instantaneously diffused through water in sufficient quantity, and immediately dissolved therein in presence of basic acetate of lead. The cell walls in this case appear to be completely torn asunder to such an extent that their saccharine juice dissolves immediately in water. There is thus no diffusion, properly so-called—simply dilution.

*Equipment of laboratory for testing beet seed-producers.*—To apply

*this process to the analysis of individual sugar beets to be used as seed-producers, a laboratory fitted up with the following appliances is required:* 1. One or more pigeon-hole cases with numbered receptacles in which the beets to be analysed are kept. 2. A drill-pumping machine, driven by hand or motor power at a speed of 2,000 revolutions a minute, to produce a very fine pulp called pulp cream. 3. One or more balances or scales sensible to 1 centigramme. 4. Tables, graduated 50 or 100 c.c. flasks, basins, funnels, nickel cups, test glasses, numbered copper forceps, filter paper. 5. A saccharimeter, working with white or yellow light according to the method of illumination adopted.

*Analytical method of testing individual beets as to their suitability for seed-producers.*—A special workman presents each beet to the drill about the upper third of its height, inclining it slightly but not pressing it. The beet, drilled from side to side, is passed to an assistant, who places it in a case, with a label bearing a consecutive number; the drill having been withdrawn, the pulp is cautiously extracted and placed in a numbered saucer, which is handed to the weigher. The latter mixes it intimately, and weighs out from it a certain quantity, which is transferred, by means of water and through a wide-necked funnel, into a graduated flask. A solution of basic acetate of lead of 30° B. sp. gr. is added to refine the juice. The volume is then made up with water to the graduated mark. If need be the froth is dispersed by a few drops of ether. The flask is shaken, then its contents are filtered, and the filtrate, clarified if need be with a few drops of acetic acid, is polarised in a 400-millimetre tube, and the direct percentage of sugar in the pulp is soon ascertained. When a large number of samples have to be tested the continuous flow tube (Fig. 4) may be used. The figures showing the percentage of sugar in each root are noted at the conclusion of each test in a special register, and opposite each analysis the weight of each beet recognised as good is placed. This weight is taken on a special balance. To avoid the fatiguing reading of the degrees, an electrical arrangement may be fitted to the saccharimeter, working to sounders of different pitch, the one grave and the other acute.

*Pellet's continuous analysis tube* (Fig. 4).—A great delay in the analysis of beets occurs in the filling, emptying and cleansing of the saccharimeters, and the time occupied by those making the readings in inserting the tubes in the instrument and taking them out again. To obviate this Pellet devised a method by which the whole of the analyses can be done consecutively in a single tube. He adapted two branches to a narrow tube 40 cm. long, one at each end, quite near the lenses. The saccharimetrical tube being placed in position, if the small rubber tube dips into a glass full of juice, and if the air be sucked out by the other tube a siphon is formed

and the glass empties itself, unless the tube be pinched to stop the current. The apparatus being arranged in this way, a small conical test glass on foot containing the juice is placed underneath the small rubber tube. The extremity of the long tube is released, and the saccharimetrical tube is filled with juice. The reading is taken, an assistant places a second test glass in place of the first, fills the siphon, and the fresh liquid takes the place of the first. A new reading is taken, registered, and a third test glass takes the place of the second, and so on. Ten thousand analyses may be done in this way daily by a staff of fifteen to seventeen persons. The estimation of the percentage of sugar in beets for seed has been brought to a high degree of perfection, combined with rapidity. In large seed establishments, like Vilmorins in France, and Klein-

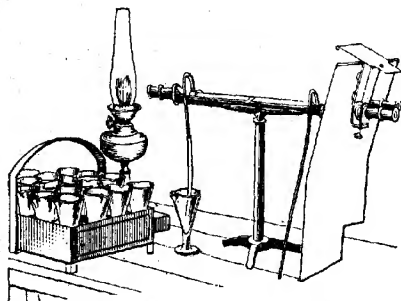


FIG. 4.—Apparatus for continuous saccharimetrical analysis of beets.

wanzleben in Germany, which make the growing of beet seed a speciality, it was necessary to find rapid methods by which one operator could analyse thousands of samples a day. These methods have just been described, and it only now remains to insist here on the importance of the analysis of the beets and the immense number that have to be analysed in a very short time in the laboratories of beet seedsmen. These laboratories have been aptly termed analyses factories, with motor-power, machinery and a large staff capable of working quickly and accurately; accuracy in that kind of work being as important as speed. Hanriot's very elegant arrangement has been installed at the sugar factory of Logras et C<sup>o</sup>. of Aulnois-sous-Laon, where 8,500 analyses per day are made with a Leudeboom drill, a knife and four Hanriot's apparatus, three of which are at work. To get the 93,618 beets necessary to produce



the seeds used in laying down the sugar beet crop grown for this firm, they expended in 1897—

TABLE VIII.—*Expenses incidental to testing beets for seed-producing purposes.*

Chemicals . . . . .	£88.58
Labour and supervision . . . . .	69.132
Light . . . . .	2.812
Paraffin oil for saccharimeter . . . . .	0.7
Coal for engine . . . . .	7.996
Loss and depreciation . . . . .	6.23

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£170.450

F. Sachs and A. le Docteur quickly accomplish a considerable number of analyses thus: The German normal weight, 26.048 grammes, is weighed out into a suitable vessel, and by means of special automatic pipettes 5 c.c. of basic acetate of lead and 177 c.c. of water are poured on to it, the vessel covered with a suitable lid and well agitated. An exact measurement is thus obtained without bothering about the air contained in the pulp. Polarisation is effected in a 400-millimetre tube. Gallois and Dupont make similar pipettes for the French normal weight. In the analysis of beets in France it is necessary to take the French normal weight for 200 c.c. and polarise in a 200-millimetre tube, as digestion is not perfect or complete with the normal weight for 100 c.c. The amount of sugar in the beet, estimated by digestion in water, is on an average  $\frac{2}{10}$  per cent. higher than by digestion in alcohol (Paternan). Ninety-eight comparative determinations were made by the alcohol and water methods, and the mean by the water polarisation was 12.98 per cent. Careful determinations were also made in comparing the alcohol extraction with the cold water digestion on a very finely divided beet pulp according to the Pellet method. The means of 28 determinations were as follows: Alcohol extraction, 12.72 per cent.; Pellet's cold water diffusion, 12.73 per cent.; ordinary polarisation in water, 12.81 per cent. Alcoholic extraction is thus preferable where scientific exactness is required.

# TESTING SUGAR CONTENT OF BEETS

21

TABLE IX.—CORRECTION TABLE FOR INCREASE OF VOLUME BY ADDITION OF BASIC ACETATE OF LEAD.

Degrees Read.	Corrected Degrees.	Percentage of Sugar Calculated on Volume.	Density of Solution.	Percentage of Sugar by Weight.	Degrees Read.	Corrected Degrees.	Percentage of Sugar Calculated on Volume.	Density of Solution.	Percentage of Sugar by Weight.
8	8.8	6.6	1.0255	6.44	16	17.60	13.20	1.0509	12.56
8.25	9.07	6.8	1.0263	6.63	16.25	17.87	13.40	1.0517	12.74
8.50	9.35	7.01	1.0271	6.83	16.50	18.15	13.61	1.0524	12.93
8.75	9.62	7.22	1.0279	7.02	16.75	18.42	13.82	1.0533	13.12
9	9.90	7.43	1.0287	7.22	17	18.70	14.03	1.0541	13.31
9.25	10.17	7.63	1.0295	7.41	17.25	18.97	14.23	1.0548	13.49
9.50	10.45	7.84	1.0303	7.61	17.50	19.25	14.44	1.0556	13.68
9.75	10.72	8.04	1.0311	7.80	17.75	19.52	14.64	1.0564	13.86
10	11.00	8.25	1.0319	7.99	18	19.80	14.85	1.0572	14.04
10.25	11.27	8.45	1.0326	8.18	18.25	20.07	15.05	1.0580	14.23
10.50	11.55	8.66	1.0335	8.38	18.50	20.35	15.26	1.0588	14.41
10.75	11.82	8.87	1.0343	8.58	18.75	20.62	15.47	1.0596	14.60
11	12.10	9.08	1.0351	8.77	19	20.90	15.68	1.0604	14.79
11.25	12.37	9.28	1.0358	8.96	19.25	21.17	15.88	1.0611	14.97
11.50	12.65	9.49	1.0366	9.15	19.50	21.45	16.09	1.0619	15.15
11.75	12.92	9.69	1.0374	9.34	19.75	21.72	16.29	1.0627	15.33
12	13.20	9.90	1.0382	9.54	20	22.00	16.50	1.0635	15.51
12.25	13.47	10.10	1.0390	9.72	20.25	22.27	16.70	1.0643	15.69
12.50	13.75	10.31	1.0398	9.92	20.50	22.55	16.91	1.0651	15.88
12.75	14.02	10.52	1.0406	10.11	20.75	22.82	17.12	1.0660	16.06
13	14.30	10.73	1.0414	10.30	21	23.10	17.33	1.0667	16.24
13.25	14.57	10.93	1.0422	10.49	21.25	23.37	17.53	1.0674	16.42
13.50	14.85	11.14	1.0431	10.63	21.50	23.65	17.74	1.0682	16.61
13.75	15.12	11.34	1.0438	10.86	21.75	23.92	17.94	1.0690	16.78
14	15.40	11.55	1.0445	11.06	22	24.20	18.15	1.0698	16.97
14.25	15.67	11.75	1.0453	11.24	22.25	24.47	18.35	1.0706	17.14
14.50	15.95	11.96	1.0461	11.43	22.50	24.75	18.56	1.0714	17.32
14.75	16.22	12.17	1.0469	11.62	22.75	25.02	18.77	1.0722	17.51
15	16.50	12.38	1.0477	11.82	23	25.30	18.98	1.0729	17.69
15.25	16.77	12.58	1.0485	11.99	23.25	25.57	19.18	1.0738	17.86
15.50	17.05	12.79	1.0493	12.19	23.50	25.85	19.39	1.0746	18.04
15.75	17.32	12.99	1.0501	12.37	23.75	26.12	19.59	1.0758	18.22

Use of the above table: To 100 c.c. of juice 10 c.c. of basic acetate of lead are added and filtered. Suppose the rotation imparted to the plane of polarisation by a column of 20 centimetres be 18°, the table gives for the corrected degree 19.8; that is the deviation that would be obtained by using a 22-centimetre tube. 100 c.c. of the juice contain 14.85 grammes of sugar, or 100 grammes of juice contain 14.04 grammes.

## TECHNOLOGY OF SUGAR

TABLE X.—TABLE FOR THE ANALYSIS OF SUGAR BEETS. FRENCH SACCHARIMETER (F. SACHS).

Sacchari- metrical Degrees.	Sugar Per Cent. By Weight.		Sacchari- metrical Degrees.	Sugar Per Cent. By Weight.	
	In Juice.	In Beets.		In Juice.	In Beets.
55	9.29	8.83	88	14.51	13.78
56	9.45	8.98	89	14.67	13.93
57	9.61	9.13	90	14.82	14.08
58	9.77	9.29	91	14.98	14.23
59	9.93	9.43	92	15.13	14.37
60	10.08	9.56	93	15.29	14.58
61	10.24	9.73	94	15.45	14.68
62	10.40	9.88	95	15.60	14.82
63	10.56	10.03	96	15.75	14.96
64	10.72	10.18	97	15.91	15.11
65	10.87	10.32	98	16.06	15.26
66	11.03	10.48	99	16.21	15.40
67	11.19	10.63	100	16.36	15.54
68	11.35	10.78	101	16.51	15.68
69	11.51	10.93	102	16.66	15.83
70	11.66	11.08	103	16.81	15.97
71	11.82	11.23	104	16.96	16.11
72	11.98	11.38	105	17.12	16.26
73	12.14	11.53	106	17.27	16.41
74	12.30	11.68	107	17.42	16.55
75	12.45	11.83	108	17.57	16.69
76	12.61	11.98	109	17.72	16.83
77	12.77	12.13	110	17.87	16.98
78	12.93	12.28			
79	13.09	12.43	0.1	0.01	Same.
80	13.24	12.58	0.2	0.03	
81	13.40	12.73	0.3	0.04	
82	13.56	12.88	0.4	0.06	
83	13.78	13.03	0.5	0.07	
84	13.88	13.19	0.6	0.09	
85	14.03	13.33	0.7	0.10	
86	14.19	13.48	0.8	0.12	
87	14.35	13.63	0.9	0.13	

N.B.—This table is calculated for a normal weight of 16.29 grammes, in supposing that the average purity increases with the richness of the beet. The determination of the density is useless. 10 c.c. of basic acetate of lead must be added, for which 1 c.c. has been deducted for the space occupied by the plumbic precipitate. The observation is made in a 200-millimetre tube. A coefficient of 95 has been allowed for the beets, although this coefficient may be too high for rich beets.

# OPTICAL VERSUS VOLUMETRIC METHODS 23

TABLE XI.—TABLE FOR THE ANALYSIS OF SUGAR BEETS, GERMAN SACCHARIMETER (F. SACHS).

Sacchari- metrical Degrees.	Sugar Per Cent. By Weight.		Sacchari- metrical Degrees.	Sugar Per Cent. By Weight.	
	In Juice.	In Beets.		In Juice.	In Beets.
30	8.15	7.64	56	14.82	14.08
31	8.40	7.98	57	15.07	14.32
32	8.66	8.23	58	15.31	14.55
33	8.92	8.48	59	15.56	14.79
34	9.19	8.73	60	15.81	15.02
35	9.46	8.99	61	16.06	15.26
36	9.72	9.24	62	16.30	15.49
37	9.98	9.48	63	16.55	15.72
38	10.24	9.73	64	16.79	15.95
39	10.50	9.97	65	17.04	16.19
40	10.76	10.22	66	17.28	16.42
41	11.02	10.47	67	17.53	16.65
42	11.27	10.71	68	17.77	16.88
43	11.53	10.95	69	18.02	17.12
44	11.79	11.20	70	18.26	17.35
45	12.04	11.44			
46	12.30	11.68			
47	12.55	11.92	0.1	0.02	
48	12.80	12.16	0.2	0.05	
49	13.06	12.41	0.3	0.07	
50	13.31	12.65	0.4	0.10	
51	13.56	12.89	0.5	0.12	
52	13.82	13.13	0.6	0.15	
53	14.07	13.37	0.7	0.17	
54	14.32	13.60	0.8	0.20	
55	14.57	13.84	0.9	0.22	

N.B.—This table is calculated for a normal weight of 26.048 grammes, in supposing that the average purity increases with the richness of the beet. The determination of the density is useless. 10 c.c. of basic acetate of lead must be added per 100 c.c. of juice, for which 1 c.c. has been deducted for the volume of the plumbic precipitate. The observation is made in a 200-millimetre tube. 95 has been taken for the coefficient for beetroot, although this figure may be in reality too high for rich beets.

The determination of the percentage of sugar in beets was for a long time made by *Violet's* process, based on the fact that when the beet pulp was treated by a dilute acid only the cane sugar is transformed into reducing sugar, which are titrated with copper solutions. By means of a small drill a cylindrical sample is taken from the upper third of the root, in such a way as to get a fair average sample of the beet for analysis. The skin is removed and the small cylinder is cut lengthwise into fine slices. These slices

are cut into finer slices, and 10 grammes introduced into a graduated flask of 100 c.c., with 10 c.c. of normal sulphuric acid and 40 c.c. of distilled water, and the whole brought to the boil. After boiling for a quarter of an hour the solution is cooled, made up to 100 c.c., and titrated with Violette's solution. This process is at the present time, according to Malpeaux, almost abandoned. The operations, he says, are tedious, the end of the reaction difficult to catch, and the results obtained generally too high. On the other hand, Ville, no mean authority, stated that the estimation of sugars by means of the cupropotassic solution is susceptible of very great precision. This precision, in his opinion, was at least equal to that given by the optical saccharimeter. In 1861 the two methods were the object of very careful comparison in Ville's laboratory on rather a large number of beetroot juices.

The following were the results obtained. Contrary to what Malpeaux says, it will be seen his results are lower than by the polarimeter:—

TABLE XII.—ANALYSIS OF BEET JUICE. COMPARISON BETWEEN OPTICAL AND CHEMICAL METHODS.

	Chemical Process. Sucrose Per Cent.	Saccharimeter. Sucrose Per Cent.
1	7.81	7.47
2	8.49	8.68
3	7.37	7.74
4	7.94	8.12
5	8.67	8.70
6	9.71	9.71

*Mode of conveyance from factory yard, silo or dépôt to the factory washing machine.*—The beets are transferred from the factory yard, where they have been stowed, to the factory proper. The first operation to which they are subjected is that of *washing*. Several different methods of transfer may be adopted, each more or less economical, more or less adapted to meet the special circumstances of individual cases. The best method is that which is the cheapest, quickest, and entails the least amount of handling and labour.

*Transfer by burrows from silo to factory.*—*Elimination of mechanical impurities.*—Shifting the beets by barrow is not practicable unless the heaps or silos are close at hand. In any case the beets must be piled with care, so as to remove all stones, straw, etc., which might cause injurious complications. To eliminate such rubbish wide pronged forks are used which can only pick up the beets themselves. Sometimes the labourers take them from the heap by hand,

This handling process is a more laborious, but often a profitable one, when the roots are very dirty and liable to cause breakdowns in the factory.

*Transfer by trucks running on tramway.*—When the silos are at a distance small trucks, running on light rails capable of being easily shifted so as to run along the open silos, are used.

*Hodgson's cable system of transport.*—Where the depôt is very far off, cable transport is adopted. This consists of a long endless steel-wire cable, stretching between the factory and the depôt, and driven by a steam engine. The cable passes over a horizontal pulley at both ends, and thus forms two parallel ropes, travelling in opposite directions. Supports, in the shape of brackets, keep the cable at a sufficient height above the ground. In this way it can pass over roads, rivers, hill and dale, throughout a course of several miles. The trucks, full of beets, hooked on to this cable, proceed, quite alone, to the factory, and are then returned empty to the depôt on the opposite rope. This system is very useful in districts with bad means of communication; moreover, the cost of freight is relatively cheap, but dearer than by locomotive.

*Feeding the washing machines.*—The cheapest way of feeding the beet washers is by *hydraulic carrier*, a narrow channel or mill-lade of about 20 inches wide, rounded at the bottom, running right through the factory yard, in which a stream of water flows rapidly. When the beets are pitched into this stream, as their density is only slightly greater than water, they are carried along with the current to the end of the channel, which dips into the washer. There is no fear of a barrow load of beets, thrown in at one point, stopping the flow of water, because at that point the level of the liquid rises, passes over the heap of beets, carrying away the top ones in its train, disintegrates the heap, and, under the ever-increasing pressure from behind, forces the individual beets to swim rapidly to the washer. The yard may be intersected by similar channels, and the labour in handling the beets is much diminished. If the different channels be completely covered with boards, the heaps may be piled above the channel itself, and by lifting the boards in rotation a single labourer can dismantle them from the one end of the heap to the other, and thus feed the washing machine at the same time. The *hydraulic carrier* is thus the most convenient and cheapest method of transport when there is a slope towards the factory. Its feed water is used for the washing machinery, for which there has to be a very abundant supply. Whatever method of transport be adopted, the beets have first to pass through one or two washing machines, to free them from the mud with which they are encumbered, a very difficult operation when they have been lifted in wet weather on heavy soils.

*The old type of washing machines.*—These consisted simply of a wrought-iron drum, pierced with numerous holes of about three-

quarters of an inch wide, revolving on a horizontal axis, beaters in the interior, and an exit for the wash water, at the opposite end to that of the entrance, caused the beets to circulate from the one end to the other. This drum dipped to half its depth into a trough of water, and thus effectively scoured the beets.

*The new type.*—*Fricourt's beet washing machine.*—The cylinder has now been superseded. Wooden blades fixed into a shaft dipping into the trough meet all requirements. The blades are arranged in a helical manner, and force the beets to advance by turning them round and round until they make their exit from the tank, pressed against the sides by the helical motion of the blades. The diameter of these machines varies between 5 and 6 feet, and they are about 20 feet long. Two of them always, and sometimes three, are

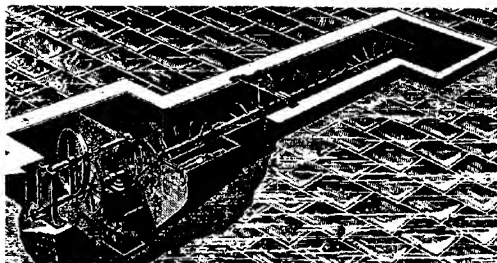


FIG. 5.—Fricourt's machinery for washing and cleansing beets and freeing them from stones.

arranged end-on to each other, the last fed with fresh clean water to thoroughly cleanse the beets.

*The stone remover.*—The beets from the last washer pass through a stone remover with iron blades in a deep tank, in which water moves at a rapid rate. If any stones be carried forward by the washers they fall to the bottom of the beck without the blades being able to discharge them, as they do not touch the bottom, the water unaided bringing the beet to the surface. The stone remover is an essential machine in all sugar-beet factories, but more especially where the beets come from pebbly clay soils. It might almost be dispensed with in the case of beets grown on soil free from stones. In Fricourt's washing machine there is an arrangement in the front—a sort of door driven automatically by gearing—which rises and falls alternately to and from a regulated height, so as to clear out any mud accumulated in the bottom.

*Loze's stone-eliminator for washing and cleansing sugar beets.*—This machine is generally placed behind the rinsing washer. It may be fixed in another position, if necessary. It acts very simply. The machine is filled with water and started. The helix, placed in a short cylinder, produces a rapid current of water, which passes

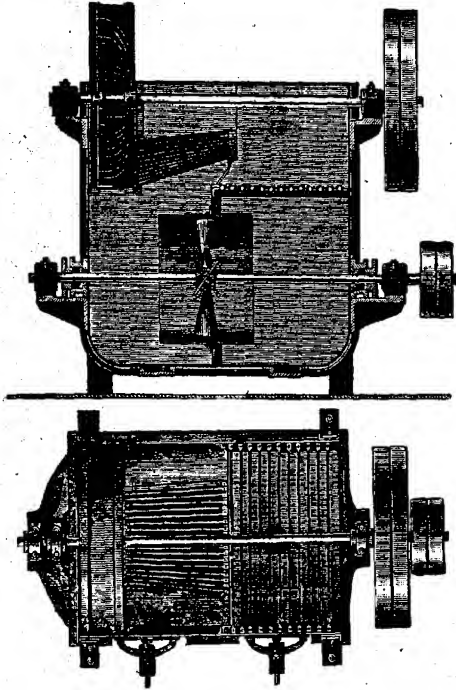


FIG. 6.—Loze's stone-eliminator, for washing and cleansing sugar beets (MAGUIN).

through a grating on which the beets fall. The latter, carried up by the current, fall on to a second inclined grating, and thence on to a small paddle-wheel, which throws them outside into the helix elevator or the succeeding washer. The stones or other small-sized heavy bodies fall through the grating to the bottom of



the machine. Large stones or other heavy bodies remain on the first grating. The machine is emptied during stoppages (meal hours). It requires but little water, because it is not renewed except at stopping time. This machine may be incorporated in the washing machine. The washed beet is then quite clean, and the next step is to extract the juice from it. Here the Excise laws of different countries come into play, and may cause slight alterations of the extraction plant.

*Weighing the washed beets.*—A necessary process in France and other countries where sugar duty is levied on weight of beets.—When the tax is levied on the finished product, i.e., upon the sugar despatched from the factory, the beet is treated for its sugar immediately it comes from the washing machines. When the tax is levied, as in France, on the weight of the raw material, the beets have to be weighed under the supervision of the Excise as they come from the washing machines. The weighing of the washed beets is so important to the manu-

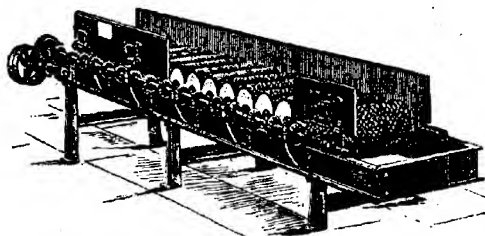


FIG. 7.—Beet wiping machine (LEFEVRE).

facturer that he ought to do it himself, when the Excise do not insist upon it, so as not only to know exactly the weight of the beets treated, but also to be able to check his stocktaking of the sugar production, and thus ascertain and trace to its source any loss in working, any preventible leakage, etc. The washed beets are therefore sometimes weighed in well-regulated factories, where the tax is levied otherwise than on the weight of the raw material.

*Drying the washed beets before weighing.*—*Maguin's shaker.*—The washed beets are, of course, wet, and must be dried, so as to avoid paying duty on water as well as on beets. The beets are therefore passed on to a shaker (Fig. 8), a metallic sieve in the form of a very slightly inclined plane, which allows the water to drain off, and animated by a to-and-fro movement, which gives it a shake at each change of direction both fore and aft. This shaker (Fig. 8) is about 3 feet wide and 16 to 20 feet long, according to size of factory. It propels the beets to the hopper of the weighing machine at such a

gradual pace that, turned round about and round about by each shake, they are sufficiently dry before they reach the end of their journey.

*Denis Lefevre's machine for drying washed beets.*—Attempts have been made to produce more perfect drying machines than those on the shaking-sieve principle. Thus Denis Lefevre's apparatus (Fig. 7) consists of a trough, the bottom of which consists of a series of parallel revolving whalebone brushes, close to one another, with their axis at right angles to that of the trough. They pass the beets along from one to another, rolling them in every direction. The beets

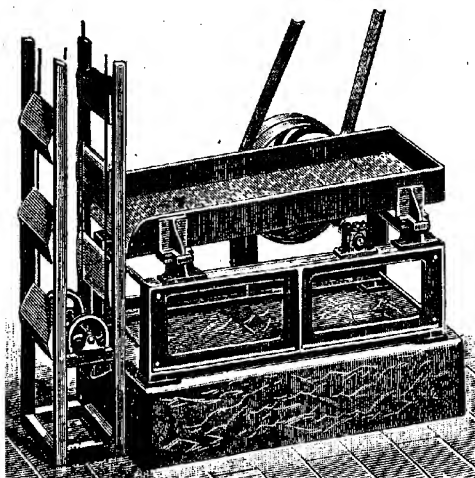


FIG. 8.—Shaker used in drying washed beets before weighing (MAGUIN).

are therefore wiped more dry than by the shaker. The upkeep of the brushes entails a certain expenditure, which is, however, compensated by the smaller amount of water which passes to the weighing machines.

*Hot-air drying ovens.*—Abortive attempts have also been made to dry beets in hot-air drying ovens.

*Elevators.*—In any case the beets pass from the drying machine to the weighing machine. The beets are transferred from carriers to washing machines, from washing machines to drying machines, from drying machines to weighing machines (appliances which cannot all

be fixed on the same floor), by elevators. There are several kinds of elevators, e.g., hemp or gutta-percha belts, inclined at an angle of  $45^\circ$ , on which paddles or cups are riveted or screwed. The beets, fed into the hopper at the bottom of the elevator, are raised to the desired height, at which point they are emptied and the string of empty cups descends to the bottom, to be recharged with beets at the hopper below. There are other kinds of elevators, on the Archimedean screw principle, revolving in a frame having the same slope as the paddle elevator. The beet is then seized in the thread or path of the screw, and is raised up with the greatest ease. But the last two classes cannot be made of any length as their slope makes them cumbersome. They are only used to raise beets a few feet. To raise beets to the top of the factory, where the weighing is usually done, vertical elevators (Fig. 9) are used. These consist of sheet-iron cups, attached at intervals to two parallel chains in place of a belt. The cups are sometimes a constituent part of the chains, held together by short chains which hook on to rings on the cups. The chain elevator has been still further simplified by inserting only a single chain between each cup. The latter glide, on each side, on a kind of wrought-iron rail screwed to the uprights of the elevator. Whatever sort of vertical elevator be adopted, the beets fall directly into the cups below, which carry them to the top, where they are shot down an inclined plane to the weighing machine.

*The requirements of the French Excise in regard to weighing machines.*—The beets must be weighed very accurately when the Excise is in charge, for neither they nor the manufacturer want their respective interests to suffer. The French Excise have imposed a series of very severe conditions and restrictions in the construction and working of the weighing machines. Firstly, that the scoop of the weighing machine cannot be emptied until the exciseman has satisfied himself that the weight is exact; and secondly, that the exciseman himself cannot authorise its discharge until the balance is quite in equilibrium. Again, it must be impossible for the manufacturer to add a single beet after the weighing has been accomplished. All these requirements have been complied with by the makers of weighing machines, in a simple and almost identical manner. A weighing machine (A, Figs. 17, 18 and 19), approved by the French Excise, consists of a scoop, with an inclined bottom fixed on an ordinary steelyard. The scoop has a hinged lid above and underneath; on the vertical face opposite to the inclined plane of the bottom it also carries a hinged door, opening from below upwards. The two doors are connected by a system of cams and levers, which do not allow of one being opened until the other is closed; so that when the lid is raised the door below is bolted, and reciprocally the door underneath can only be opened when the lid above is bolted. Filling and emptying are thus quite distinct; not

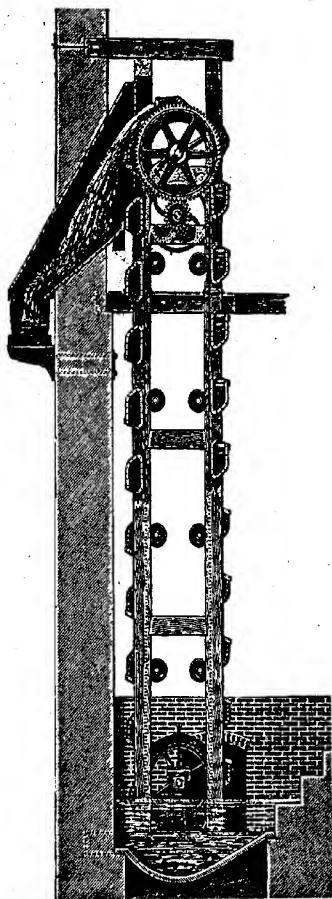


FIG. 9.—Vertical elevator for raising beets (MAGUIN).

a single beet can enter the scoop before it has been completely discharged. Again, the framework of the steelyard, which supports the pans itself, carries an œillet to hold a bolt, and this bolt, which is fixed, cannot enter the œillet until it is quite opposite it, *i.e.*, when the steelyard is in equilibrium and when the scoop holds the exact weight of beets, which is represented by the weights placed in the plate of the balance. When this occurs, the exciseman, who holds the lever of the aforesaid bolt, can push the latter into the œillet. But the lever during this movement acts upon another lever, which undoes the bolt of the lower door of the scoop and bolts the upper door, so that, so long as the balance is not in equilibrium, it is impossible to open the lower door, and it is only when the exciseman has brought his steelyard into equilibrium that it is possible to empty the scoop. When the latter is discharged the exciseman restores the lever to its place, fixes the door underneath, and allows the upper door to be opened. Each time the exciseman pushes his lever this movement puts an automatic register in motion. Every time he opens the bottom door he puts a second automatic register in motion. Each time that the supervisor passes, these two registers should correspond exactly, with the exception of a false manoeuvre, the details of which are registered in the books.

*The system works without any great friction.*—This method of weighing works well, and causes little friction between the Excise and the manufacturer. But it is rather complicated; if anything gets out of order, it is a bad business. But this must be borne with, as it is necessary to collect the tax, and the present tax on beets is held in France to be the most equitable of any.

*German rules as to weighing not so strict as the French.*—In Germany the tax on beet was formerly imposed as now in France. The steelyards were more simple, and Excise control was not nearly so strict.

*The old French weighing machines assimilated to the new régime.*—However, in certain French factories where weighing was in vogue before 1884, the year when it was rigorously enforced by law, the existing state of matters was respected and regulated. Thus we come across weighing being done on tramway trucks, the conditions being that the latter enter a completely closed cage before being weighed.

*Extraction of beet juice by pressure.*—As this process is now a thing of the past, we need not occupy space describing an obsolete method.

## CHAPTER II.

### DIFFUSION.

*HISTORY.—The early attempts of Mathieu de Dombasle.*—About 1830 Mathieu de Dombasle (1777-1843), a celebrated French agronomist, who not only in his day made improvements in many agricultural implements, but who was also one of the original creators of the beet-sugar industry, introduced a maceration process for extracting its juice from the sugar beet without pulping, rasping or grating the beet, and without hydraulic presses. The beets were cut into thin slices by a rotary machine. The slices were then transferred to the No. 1 of a series of casks arranged in the form of a battery, the juice of No. 1 being run on to No. 2. The beet slices were macerated in No. 1 for about an hour with about their own bulk of water, at a temperature of about 212° F. After this treatment the liquid—having now acquired a density of 2° Baumé, sp. gr. 1.014—was run off into No. 2 containing fresh beet slices. From the No. 2 it was run into No. 3, and so on until it had passed through the No. 5, charged in the same way, when, its density having reached 5½° Baumé, sp. gr. 1.040, it was suitable for 'defecation. No. 1 was thus charged with hot water, and No. 5 yielded a juice suitable for further treatment. To prevent cooling, it was reheated in its passage through the casks, and the maximum amount of sugar possible was thus extracted. The exhausted slices scarcely contained any sugar. But juice obtained thus, although transparent and requiring little lime for purification, was liable to ferment, or, owing to the dilution water, it was difficult to granulate. Hence the process was generally abandoned, as the juice obtained by it could not be successfully treated by the method then in vogue for pressed juice, and because the exhausted slices were too wet for cattle-feeding.

*The double-carbonatation process clears the way.*—Matters remained so for some twenty years, until 1849, when Rousseau made a great step in advance by indicating a practical process of successfully treating beet juice, no matter of what nature or how obtained.

In 1849 Perier and Possos in France and Jelinek in Austria put

the finishing touches to *Rousseau's* process, and their system of treating the juice was termed the *double-carbonatation* process.

*Final triumph and economy of the diffusion process.*—Owing to the progress then made it was found practicable to treat the maceration juices obtained by the method of Mathieu de Dombasle. Moreover, about 1860, this process was revived by *Robert*, a sugar manufacturer of Seelowitz, Austria, who, out of the rudimentary appliances of Mathieu de Dombasle, evolved quite a new industrial system of treating the juice, called the *Diffusion Process*—a process destined to revolutionise the whole beet-sugar industry. He not only made De Dombasle's process practicable, but even economical.

*The result of the action and reaction of the different improvements in the treatment of the juice.*—The diffusion process, therefore, says Horsin-Deon, only became practicable after the methods of working had been improved. Again, these methods could not have themselves been improved had it not been as a result of the invention of new machines. It has, therefore, been well said that the majority of innovations in sugar manufacture are connected together by a common bond. As chemists of high standing, engineers and manufacturers of great merit, have each and all tried all the improvements which their fertile brains could suggest, it is rare to find a real novelty in recent inventions. But those inventions, which were formerly impracticable, have only come into use in virtue of the immense progress in engineering in virtue of improved plant. Nevertheless, continues Horsin-Deon, each invention which sees the light of day brings its seal of novelty to the cause even of that progress from which it flows, even although it be only an old method revived. In this way was diffusion born and developed in our own days, thirty years after it had been forgotten. In this way we have seen carbonatation regenerated, after the invention of filter presses and twenty-five years after Kuhlmann first thought of it; and Steffen's process of cleansing *masses cuites* adopted sixty years at least after the American *tigers*. Who knows what use our sons may make of our modern researches, often now without practical application? Happy are they who know how to profit by the knowledge of their predecessors! They are few and far between. This explains the numerous actions of nullity of patent, which have stopped somewhat the inventions of our time; for why should one work if the laws insufficiently protect those who spend their intelligence and their money in the cause of progress. There have been diffusion lawsuits, carbonatation lawsuits, filter-press lawsuits, evaporation lawsuits, lawsuits about everything. This is history, and it may be beneficial to mention in passing, in a *résumé* like the present, the difficulties in the genesis of the sugar industry. May our successors be more fortunate! (Horsin-Deon).

*The scientific principles on which diffusion is based.*—Although

apparently rather complex, the principles underlying diffusion are very simple. When a thin well-cut slice of beet comes in contact with water an exchange of liquids is made between the saccharine juice contained in the beet cells and the water in contact with the slice of beet, according to what is called the laws of osmosis or dialysis.

*Osmosis and dialysis.*—If we run in a solution of common salt in water into a bladder and tie it up securely, and suspend the bladder in a vessel containing distilled water, we will find after a certain time that the water outside the bladder is as salt as that inside the bladder. Equilibrium tends to establish itself, and at the end of a certain time—of greater or less duration—the two liquids acquire the same degree of concentration. Now, if instead of a solution of salt we take a weak solution of glue—so weak, in fact, that it cannot set into a jelly—practically none of the glue permeates through the bladder into the water in the outer vessel. Again, if a mixture of solutions of glue and salt be run into the bladder and similarly treated, the two substances will be found after a time to have separated, the salt having passed through the bladder into the outer vessel, and the glue remaining behind inside the bladder. A great number of substances, especially those capable of assuming the crystalline form, behave in a similar manner to salt, and pass through the bladder, e.g., sugar. This can be demonstrated in a somewhat similar manner to our experiment with salt. If we securely cover the end of a piece of rather wide glass tubing with bladder, and fill it with a solution of sugar, and then close it at the other extremity with bladder in the same way, and immerse the tube in distilled water, the bladder closing the ends of the tube will, after a certain time, be found to be distended or bulged outwards, owing to the pressure of the water which has been gradually absorbed from without inwards. The water in the outside vessel has, moreover, acquired a sweetish taste, showing that a portion of the sugar solution has passed through the bladder from within outwards.

*Osmosis differentiated into endosmosis and exosmosis.*—This diffusion of fluids through a closed membrane is called *osmosis* (Greek, *osmos*, an impulse or pushing), the passage of the fluid from without inwards being differentiated as *endosmosis*, and the passage of the fluid from within outwards as *exosmosis*.

*Crystalloids and colloids.*—Again, substances, the solutions of which pass through the animal membrane, like salt and sugar do, are termed *crystalloids*, whilst those bodies, like glue, which do not pass through the membrane are termed *colloids*. This differentiation of substances into *crystalloids* and *colloids* is more relative than absolute, because crystalloids themselves pass through porous membranes at different speeds, and even *colloids* are not absolutely deprived of this property. It is more correct to say that there is



an ascending and descending scale of the osmotic properties of bodies, in which certain crystalline substances are placed at the top, and certain amorphous bodies at the bottom.

*Differentiation between osmosis and simple filtration.*—But a membrane may be permeable to osmotic phenomena without acting as a filter. Filtration and osmotic phenomena must not be confused. Again, a membrane may be permeable to gases without being permeable to liquids. (Gases are subject to the same laws of osmosis as liquids.) India-rubber, for instance, is permeable to gases, whilst it is quite impermeable to liquids, or at least to those which do not act upon it chemically. Parchment paper, likewise, is permeable to liquids, but nevertheless cannot be used for filtering purposes. The same laws apply to the membranes of vegetable cells which are essentially favourable to osmosis, for osmosis is one of the vital principles of plant life. When two liquids, therefore, of different densities are separated by a porous membrane, there is intercommunication between them, and a passage of the two liquids respectively through the membrane, in opposite directions, and the speed of the passage is inversely proportional to the density of the liquid, i.e., the less dense liquid will pass through more quickly. If one of the two liquids in solution be sugar, the saccharine fluid will pass through the membrane, in one direction, whilst the pure water passes, in an inverse direction, much quicker than the saccharine fluid passes outwards. The sugar content of the saccharine fluid in the beet cell, therefore, becomes less and less, whilst that in the exterior fluid increases more and more, and the phenomena of diffusion cease when the sugar content of the fluids is the same on each side of the membrane or diaphragm. Equilibrium has been established. Now, if at this point the exterior saccharine fluid be run off and replaced by pure water, osmosis starts afresh, and by renewing the water indefinitely the sugar content of the original liquid inside the membrane is finally reduced to nil. If a current of pure water, therefore, be made to circulate in the outer compartment, the whole of the sugar contained in the interior compartment will gradually pass through the partition. The cellulose, which forms the partition of vegetable cells, behaves like a permeable partition to *crystalloids*. But it only allows gelatinous substances, e.g., albuminoid bodies, to traverse it with extreme difficulty. The principle, therefore, of the diffusion process of sugar extraction rests on the phenomena of osmosis. The actual operation is carried out by causing a current of water to circulate round the slices of beet until they are exhausted of all the sugar contained in their cells.

*The diffusers, their general arrangement.*—*The sugar-beet diffuser.*—A diffusion battery consists of a series of eight to fourteen cylindrical vessels arranged consecutively, and called *diffusers*. They communicate with each other by piping, so that the juice, issuing

from the bottom of one diffuser flows into the next from above. The current may be reversed by taps if need be, so as to pass from top to bottom of the diffusers, instead of from bottom to top, or the juice may be heated during its passage from the one battery to the other.

*The calorisor.*—A steam reheater, or *calorisor*, keeps the liquid always hot. Again, taps are so arranged that water may be run into each diffuser, instead of juice. There is also a tap for running off the liquid after the beets have been exhausted.

*Charging and discharging doors.*—The diffusers have a top door for charging them with fresh beet slices, and a bottom door through which the exhausted beet slices are discharged.

*Theory of the working of sugar-beet diffusion batteries.*—Supposing all the diffusers are charged with fresh beet slices, there will still be a certain amount of vacant space between the slices. When enough water is run into the diffuser to occupy this vacant space, the diffuser will then contain about equal weights of water and beet slices. Not only so, but the space occupied by each is also almost identical, the density of the beet being only slightly above that of the water. If the first diffuser of the series be now charged with water, *osmosis* at once starts to act in the cells of the beet so as to cause a certain proportion of the sugar which they contain to pass into the water. The batteryman, however, does not wait until equilibrium in density is established between the saccharine fluid in the beet cells and the exterior saccharine liquid, as that would take too long. After a few minutes contact, however, there is no very great difference in density between the two. The liquid from the first diffuser, such as it is, is then run into the diffuser next to it, being reheated in its passage. The saccharine liquid is now in contact with fresh beet slices, whose juice is of greater density than its own, *osmosis* is again energetically started; it thus soon becomes still more highly charged with sugar, until, in fact, the densities are nearly equal. The saccharine liquid is once more run into a fresh diffuser, and the same interaction of fluids takes place, and the same operations are continued until the density of the liquid is only slightly inferior to that of the primitive juice. The *osmotic* action is then almost nil.

*The superior quality of diffusion juice.*—Supposing that the passage of the liquid through the diffusers is stopped at No. 6, after which it is sent to other departments of the factory for further treatment. The sugar content of the *diffusion juice*, as it is termed, is now so great that its density is approximately the same as the *primitive juice of the beet*. But the two juices are not identical by any manner of means. In addition to sugar the beet also contains salts in solution, which have passed through the membrane of the beet cells simultaneously with the sugar. But—and herein lies one of the many

advantages of the diffusion process—the diffusion juice contains little or no colloids, such as vegetable albumen, pectose, etc., consequently, it

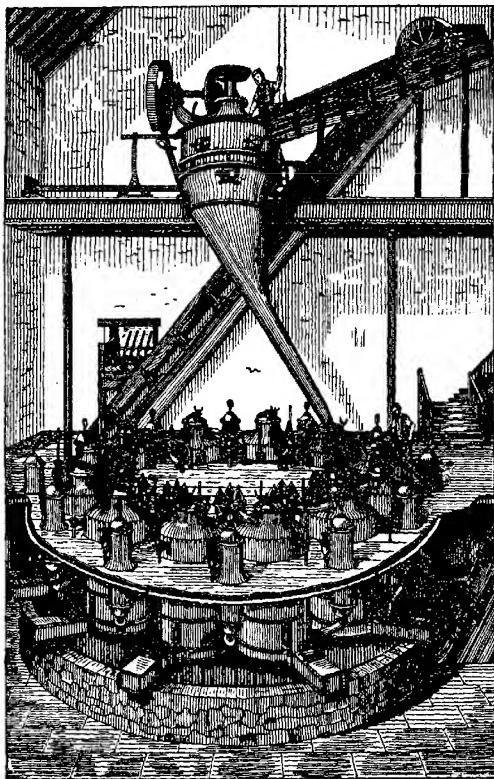


FIG. 10.—Circular diffusion battery for extracting the saccharine juice from beets by diffusion, showing inclined beet elevator, slicing machine, revolving funnel feeder, diffuser, staging, and pit into which exhausted slices fall.

is much more pure than real beet juice, because with both hydraulic and continuous presses the real beet juice is obtained by the more complete shredding of the cells by the teeth of the pulper or grater machine, so that it includes not only the crystalline sugar, but all the colloid bodies present in the juice. Diffusion juice is therefore far superior, so far as purity is concerned, to that obtained by pressing the grated or rasped pulp.

*The sequence of diffusion operations.*—Having now obtained a clear idea of the way in which the sugar is extracted from the beet slices in each diffuser in succession, and of the advantages such juice possesses over pressed juice, let us still further follow the sequence of operations. We left the diffusion battery in the following condition: No. 1, diffuser contained half-exhausted beets; No. 2, quarter-exhausted; No. 3, one-eighth exhausted, and so on. If pure water be now run into No. 1, and circulated in the consecutive diffusers as before, No. 1 will be three-quarters exhausted, No. 2 will be half-exhausted, and so on, until we stop at No. 7, by which time the diffusion juice thus obtained is quite as dense as that obtained in the first cycle of operations. Similar phenomena occur in the next and succeeding cycles; thus if water be once more run into No. 1 it will after that be seven-eighths exhausted, and this time the juice is circulated to No. 8 before running it off for treatment, and so on until the last diffuser of the battery is reached.

*The diffusion extraction process a continuous one.*—This continuous sequence of operations can be carried on uninterruptedly or simultaneously so that the "cells"<sup>1</sup> are always filled with liquid. The juice of the one diffuser pushes the juice of the other in front of it out of the one diffuser into the other. Water under pressure is run into No. 1, which drives in front of it the juice contained therein; the juice of No. 2 displaces that of No. 3, and so on, until the last diffuser is reached, from which it escapes into a gauged reservoir, called the measuring tank, in which only that amount of juice which corresponds with the capacity of the cell is collected each time that the operation is repeated. The sugar content of the juice as it is drawn off will be greater or less, and the exhaustion of the beet slices in No. 1 more or less perfect according to the volume drawn off and the interval between each repetition of the process.

*Calorisators, their functions and action.*—As there is a calorisor between each "cell," and as increased temperature shortens the osmotic action and consequently facilitates exhaustion, a temperature of 75° to 80° C. (167° to 176° F.) is maintained in the cells.

The greater the number of cells in operation the more effectual

<sup>1</sup> Each single diffuser is called a "cell" of the battery, but the term diffuser is retained up to here to avoid confusion with the "cell" of physiological botany, to which we have had so often to refer.

will be the exhaustion of the beets, as a greater quantity of pure water will have to be run into No. 1.

*Comparative exhaustive effect of batteries with (a) a larger and (b) a smaller number of cells.*—Of two batteries with a different number of cells, the battery with the larger number will do the greater amount of work, on account of the larger volume of water run into No. 1. The same result may be obtained by diminishing the time occupied by diffusion in each of the batteries. But then it would be necessary to heat a short battery to a greater temperature than a long battery, to the injury of the juice, because *osmosis* is more energetic when hot than when cold; hence the colloid bodies dialyse more easily when hot than when cold, and thus the juice is more impure.

*The general routine of diffusion work.*—Suppose all the "cells" of the battery to be charged, and that No. 1 has to be emptied. This is done, and it is refilled with fresh beet slices. No. 2 cell now becomes No. 1. The next run, the new No. 1, is emptied, and the original No. 3 now becomes No. 1, and so on.

To work systematically and economise time in the rather protracted operations of filling and emptying, there is always, in each battery, a "cell" being filled at the tail end and a "cell" being emptied in front, and it is the "cell" following the latter which is under hydraulic pressure. When it comes to the last "cell" instead of running in the juice from above downwards on to the fresh beet slices, it is run in from below upwards. The object is to lift up the slices and mix them with the water or to mash them. Were the water to be run in from above, the slices would be piled in the bottom, and admixture of beet slices and juice would be imperfect. It is the juice of the last "cell," after mashing, which is sent to the factory as the most concentrated, and it is the juice following which is used to mash the last "cell".

*Method of working diffusion process with short water supply.*—With a short water supply, instead of pushing the juice of the last diffuser out by water reservoir pressure, or by hydraulic pressure, compressed air is used for the purpose. Three-quarters of the water generally lost in emptying the last diffuser is thus saved.

*Beetroot slicers.*—The principles on which they should be constructed.—The shape of the slices into which beets should be cut before being run into the cells can be very aptly defined by comparing them to the slices of vegetables used in making soup.

*The success of the diffusion process depends on the size and shape of the slices.*—The beet slice should be regular, long and well cut. These are the three important points. The size of the section varies with the nature of the beet. There is every advantage in cutting the slices as thin as possible—say one-fifth to one-sixth of an inch square. Should the beet be tender or softened by drying or frost, large-sized

slices should be made. On the selection of the size of the slice often depends the successful working of the battery, especially the thorough exhaustion of the slices, *i.e.*, the quantity of sugar lost by the factory in the residues. Since as little as possible of the sugar contained in the beet should be lost, the size of the slice is important. The shape of the slice also greatly influences the yield; rectangular slices become glued together and obstruct the circulation of the water. By imparting to them the bent shape of the tiles used to cover the ridge of a roof, a large number of channels are created in the mass of slices which greatly facilitate the circulation of the water over the whole surface of the slice. These different shapes and sizes are got by imparting an appropriate shape to the root-slicers, and arming them with good cutting knives acting at sufficient speed to make a neat cut. To attain these different results, all efforts have been concentrated to impart to the root-slicers the most desirable shape.

*Description of a typical beet-slicer.*—The revolving plate, the knives and knife-holders.—The essential organ of a beet-slicing machine is a circular horizontal plate revolving rapidly round a vertical shaft. This plate is pierced with apertures in which knives, arranged like the cutting edge of a plane, are inserted. If the beet, therefore, comes in contact with this revolving plate the knives will plane the beet, and the slices thus cut off fall beneath. Moreover, if the knives have an appropriate shape the shavings or slices cut off will have the desired shape just described.

*The feeding of the beets into the hopper, and the way in which they are cut.*—The revolving plate is enclosed in a frame surmounted by a hopper and shaped underneath like a large funnel. The beets are fed into the hopper, and, as the latter is of a certain height, 20 to 40 inches, the weight of the beets above, pressing on those below, and which rest immediately above the plate, acting as an automatic pusher, causes the latter to be caught by the revolving knives and thus to be cut into slices of the desired size and shape. The beet-slicer is thus nothing more than a special adaptation of the turnip-slicer so long extensively used in Britain for cattle-feeding purposes.

*The fitting of the knives into the knife-holders and the knife-holders into the plate.*—There are 8 to 10 apertures in the plate, into which the blades are fixed. The knives are not fixed directly into the plate. They are mounted in movable, easily changeable frames, called knife-holders, and it is these knife-holders which rest in the apertures of the plate.

*Removing blunt knives and inserting a sharp set.*—As the knives soon become used up, there is always a complete complementary set, adjusted beforehand, in reserve, so that when the knives at work do not cut properly, the knife-holders with the blunt blades are removed bodily, and immediately replaced by fresh sets. The change

is thus effected very rapidly, for the construction of the apertures is such that the change involves no difficult operation, the knife blades simply fitting into the grooves which hold them fast during rotation. They are, in fact, kept in place by centrifugal force.

*Gearing.*—The plate is driven by conical gearing acting on its shaft, the gearing being driven by belts and pulleys. The shaft rests in a socket, and is kept in the vertical position by strong plummer-blocks surrounding it on the top so that it cannot shift.

*Diameter of the plates, and speed of revolution.*—The diameter of the plates is very variable—the average is 5 feet—some are 3 feet and others over 6 feet. Plates of great diameter are much in vogue in Austria. But as the speed of the knives ought always to be the same, plates of large diameter must revolve more slowly than smaller ones. Plates of 5 feet in diameter make 100 to 120 turns, so that 6½-feet plates need only make 60 to 90 turns for their circumference to have travelled the same space in the same time as the 5-feet plates.

*The different kinds of knives in use.*—Knives may be divided into three classes: (1) *Naprawil knives.*—The first, the oldest, called Naprawil knives, make rectangular slices. They consist of a straight cutting blade surmounted at intervals by cutting ridges, which divide into small sections the slices cut off by the knife. (2) *Goller knives.*—The second class of knives, known as Goller knives, make triangular slices. They are steel blades  $\frac{1}{8}$  to  $\frac{1}{4}$  inch in thickness, cut in the body in a zigzag form at an angle of 60°. They are also made of wrought iron bent into an undulating form having the same profile. The beet can thus be cut into *triangular* slices. But the form of the slice is quite irregular, because, when the knife passes through the beet, it leaves its triangular mark upon it. When the next knife comes into play it therefore cuts the ridges of the triangle, forming an irregular-shaped slice. This is bad, because the new slice has not the thickness requisite for good diffusion working. The thin slices are exhausted sooner than the thick ones, and the exhaustion is thus altogether imperfect and incomplete. (3) *The roof-ridge-tile-shaped knives.*—The third class of knives, the most extensively used, combining the principles of the Goller and Naprawil knives, are the roof-ridge-tile shape. They have the same profile as Goller's knives; only on the upper part the summit of the angle carries cutting ridges like the Naprawil knives. When a knife has passed, leaving the mark of its shape on the beet, when the next arrives, if it passes into the same furrow, it therefore lifts a roof-ridge-tile-shaped slice of perfect shape, and sharply cut on all its faces. Taking care to mount the knives so that their blades are correctly placed one behind another, it is possible to get perfectly regular slices. Yet many slices are still defective, but less so than with the Goller knives.

*Knife-holders.*—The knives are mounted in a knife-holder like the chisel of a plane. The slope of the knife and the length of steel which overlaps the plane of the knife-holder is varied, so that the knife may catch more or less as the slice is to be larger or smaller.

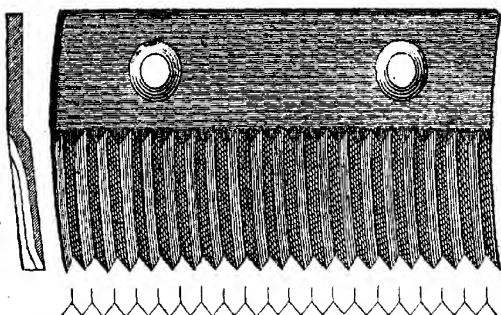


FIG. 11.—Knives (MAGUIN).

*Method of sharpening and tempering the knives.*—The knives are sharpened on very hard steel discs which revolve rapidly on their axis, and the circumference of which is dressed like a file. The profile of the bottom of the blade is given to the circumference, and

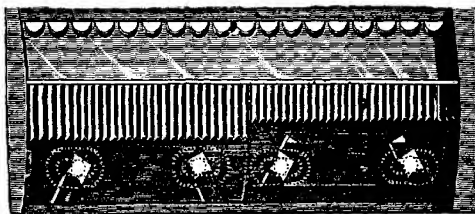


FIG. 12.—Knife-holder, non-stone remover.

it is enough to place the knife in front, firmly held in a clip which guides it, and to press lightly with the hand, to sharpen its cutting edge. There are also similar discs for sharpening the cutting edges of the ridges. A knife is thus sharpened very quickly. The sharpening is finished by files, also of the desired profile or shape.



*Hardened untempered steel, and tempered steel knives.*—Some knives are made of hardened non-tempered steel, and are sharpened immediately they are taken out of the beet-slicer. These are the class of knives most generally used. Some, made of tempered steel, require softening before sharpening and tempering afterwards. It is a big job, which requires great care to do it well, and consequently

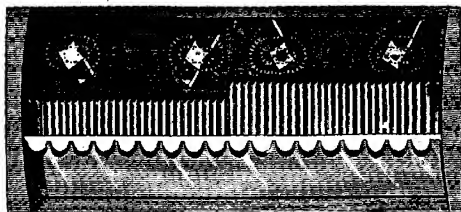


FIG. 13.—Knife-holder and stone remover.

necessitates a special smithy and a skilful, experienced smith; it is therefore practised in but few factories. The knives in current use only have been mentioned. There are others derived from the three described, only differing in unimportant details.

*Fontane and Rassman's beet-slicer.*—Another form of root-slicer invented simultaneously by Fontane in France and Rassman in Germany, of a different shape, has had indifferent success.

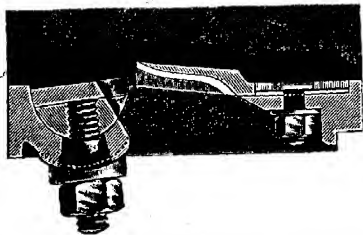


FIG. 14.—Knives and knife-holders of the beet-slicers (MAGUIN).

*Description.*—It is a vertical cylinder pierced by eight to twelve apertures, in which the knife-holders are fixed. The cylinder has the height of two superimposed knife-holders.

*Method of working and the function of its parts.*—In the axis of the cylinder a shaft, carrying two blades, which sweep the knives in turning, revolves very rapidly. The beets fed into the cylinder

are seized by the blades, and the centrifugal force presses them against the sides of the cylinder. Carried round by the blades, the roots are cut by the knives and expelled as very uniform slices.

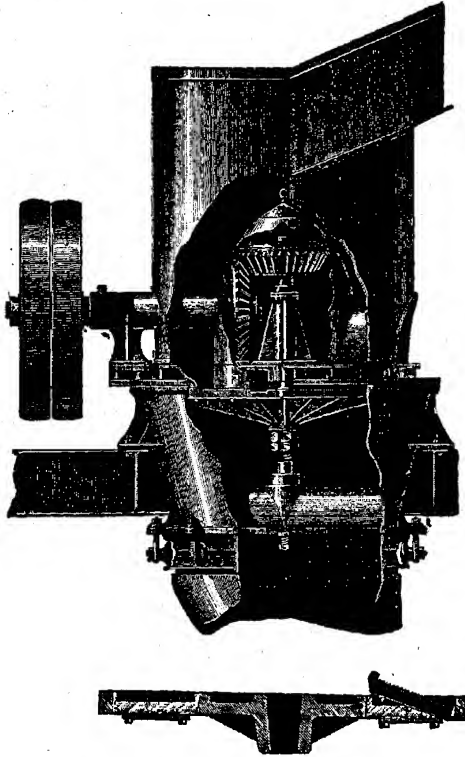


FIG. 15.—Beet-slicer (FONTANE AND RASSMAN).

*The horizontal plate machine still preferred.*—Other root-slicers on the same principles have since appeared, but the horizontal plate machine is still preferred by manufacturers. The following recent machine, however, well merits a special description :—

*Maguin's rotary drum root-slicer.*—In this machine all the knives are parallel. The knives revolve in every point at the same speed. They are close together, and the root is held until cut. These conditions are obtained by the following arrangements. The drum, 1.2 metres (47 inches) in diameter, .33 metre (13 inches) of interior cutting width, mounted on a horizontal axis, carries knife-holders to the number of eight all over its surface. Each knife-holder is furnished with six parallel knives, in pairs, with an instantaneous

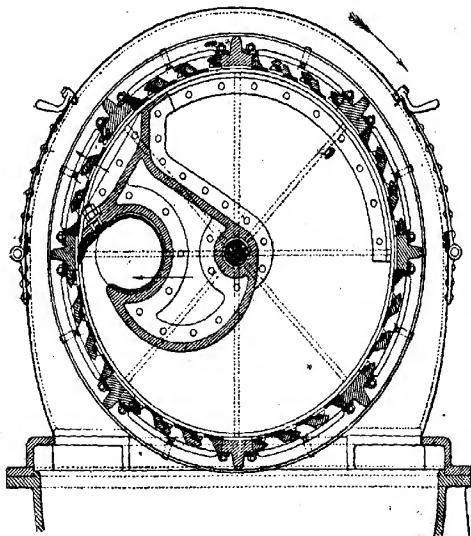


FIG. 16.—Rotary drum root-slicer (MAGUIN). Cross section.

regulating plate in front of each row of two knives. These eight knife-holders therefore represent twenty-four ordinary knife-holders, and carry forty-eight knives of 165 millimetres ( $6\frac{1}{2}$  inches) in length in twenty-four parallel rows of two knives. Being parallel, these twenty-four rows of knives go at the same speed. The beet, as soon as the half of its diameter is introduced, is carried by the circular part of the drum to a more and more restricted section formed by a rigid piece of cast iron in the form of a hook. The beet is carried in by the knives, which, being very close, slice it,

following the same edge, the more so as it is constantly kept in its position and pressed against the cutting surface. At the end of the hooked piece of iron is a cavity forming a stone-eliminator, in which pieces of beetroots, hard substances, stones, pebbles, etc., are lodged without doing serious damage. The stone-eliminator is simply and

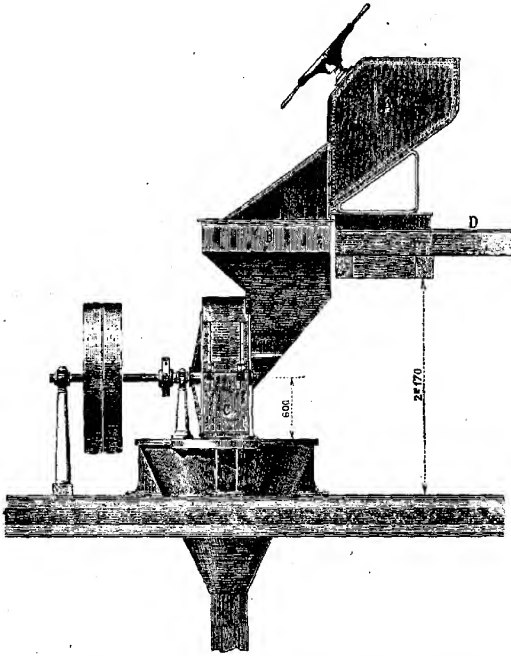


FIG. 17.—Rotary drum root-slicer mounted on body of old root-cutter (MAGUIN).

rapidly emptied by a suitable arrangement. Easily-wrought rods and a discharge door enable the beets and other bodies caught by the hook to be removed. The knife-holders are replaced as rapidly as in an ordinary root-cutter. The output of this machine, owing to the number of knives which it contains, and the speed with which it can be driven, is very considerable. The knives, moreover, are

parallel, so there is nothing to fear from the effects of centrifugal force. At 60 revolutions it easily puts through 20 tons an hour, and

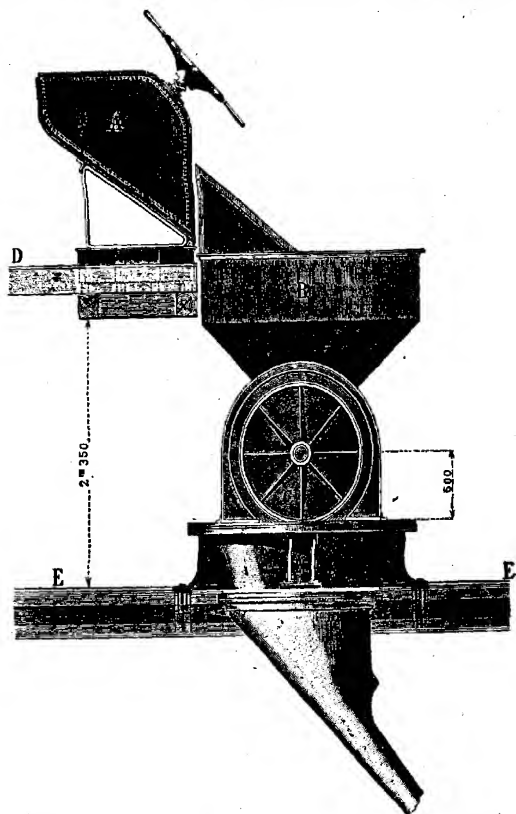


FIG. 18.—Installation of two rotary drum root-cutters on body of ordinary root-cutter (MAGUIR). Profile.

it is perhaps capable of revolving at 80 revolutions a minute, and more if necessary. At 80 revolutions it does not reach the speed of

the old root-cutters, and it only uses up 70 per cent. of the force necessary to work one of the latter machines. Fig. 17 shows the installation of one of these machines mounted on the existing body of an old root-cutter, with its special hopper and weighing machine. It requires no cog-wheels, and leaves a large space around it for manipulating the knife-holders. Figs. 18 and 19 show the profile

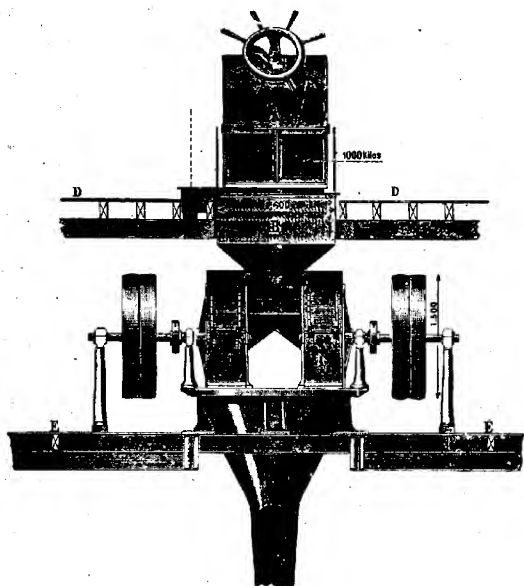


FIG. 19.—Installation of two rotary drum root-cutters on body of ordinary root-cutter (MAGUIN).

and the front of an installation of two drum root-cutters mounted on the existing body of an ordinary root-cutter of 6½ feet in diameter. By this convenient arrangement the production of slices may be doubled if the machines work together; and continuous working is guaranteed if worked alternately, an advisable arrangement for factories which treat more than 400 tons of beets in the twenty-four hours.

## BEETROOT PULP SAMPLER.

This apparatus is fixed in the chute which leads from the root-cutter to the diffuser and automatically samples the beet slices as they enter the diffusion battery. It consists of an inclined plane *b*, placed below the level of the root-cutter, pierced by a longitudinal hole *d*, in which a round stone *g* carrying a strap *a* provided with spikes *c* passes. The band *a*, passing over the stone *C*, is prolonged over a second stone *C*<sub>1</sub>, which imparts to the belt the motion which it receives direct from some transmitted power, if the apparatus be fixed on a root-cutter, the chute for the pulp of which is

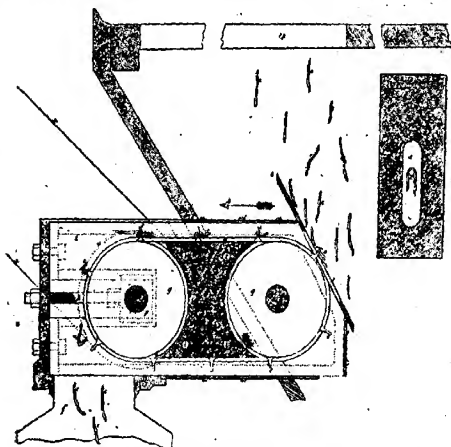


FIG. 20.—Apparatus for sampling beet slices as delivered to diffusers.

fixed, or if the fixed funnel be high enough to lodge the apparatus. If the chute be used to feed a circular diffusion battery working immediately below the plane of the root-cutter, the sample may be driven by clock-work. The distance between the stones *C* and *C*<sub>1</sub> being very short, the stone *C*<sub>1</sub> is fixed on a suspension platform, which always gives enough adherence to the band *a*. The root-cutter being at work, and the band *a* also, the apparatus works as follows: The slices fall from the plate of the root-cutter, and some of them, falling on the inclined plane *b*, are carried on the spikes on the belt *a* and fall into a reservoir *f*, which preserves them until they are required for analysis. The whole of the apparatus is

enclosed in cast-iron plates *l*, so as to hinder other slices than those falling on the inclined plane being drawn into the reservoir. The longitudinal opening of the inclined plane being very narrow, few slices are carried away at the same time. The sample thus taken enables the exact amount of sugar going into the battery to be ascertained. The percentage of sugar is multiplied by the weight of beetroots received at the factory. The difference between the sugar despatched from the factory plus the stock and the sugar received into the factory in the form of beets gives the loss on working.

*Diffusion batteries.*—Detailed description of a "cell," size and

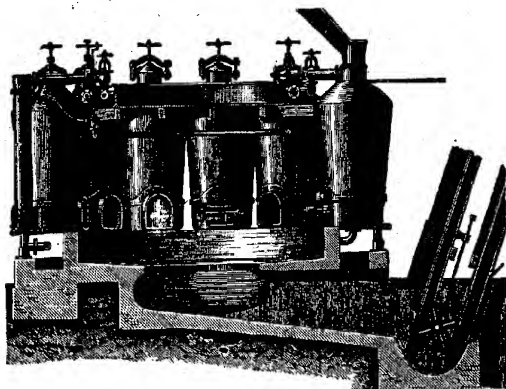


FIG. 21.—Circular diffusion battery, with inclined elevators removing exhausted pulp.

*shape.*—The "cells" (Figs. 21, 23 and 24) are vertical cylinders, the capacity of which varies from 1 to 6 cubic metres. In France they are made of wrought iron of 3 to 4 cubic metres capacity, with a height double their diameter.

*The charging manhole on the top.*—They terminate at the top in a short cone bearing a cast-iron collarette (on which is the juice circulation tube) closed from above by a door which is displaced horizontally round a turning pivot (Fig. 23) or upwards (Fig. 24), and fixed in position by a strong screw supported by a stirrup. Through this manhole, 24 to 32 inches wide, the diffuser is charged.

*The shape of the bottom and its door.*—The bottom of the cell varies in shape with the position of the door for discharging the exhausted



pulp. This door may be placed in the side (Fig. 21) or underneath (Fig. 23), or in an intermediate inclined position. When the door is on the side the bottom of the diffuser is of *wrought* iron, and almost flat.

*The manipulation of the bottom door.*—When the discharge exit is underneath, the best position, the door embraces the whole bottom of the cylinder, which then ends in a cast-iron armature which supports the door. This may open by horizontal displacement, like the door on the top, or from below downwards, round a hinge. It is balanced by a counterpoise (Fig. 24) in either case, so as to

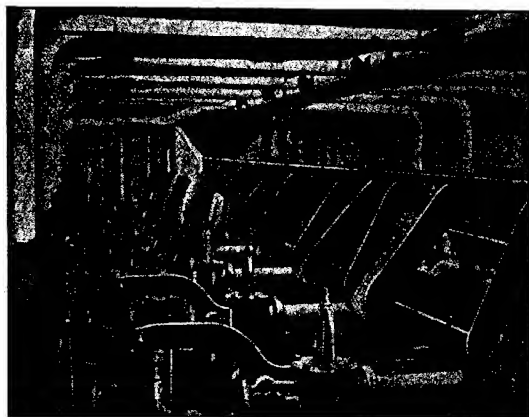


FIG. 22.—Showing the manner in which the beet slices are fed into the diffusers of line batteries (CAIL).

enable it to be handled, as it is very heavy. It is held in position by strong bolts or screws. The bolts and screws are screwed on and undone by a shifting spanner, which guarantees their good working.

*Inclined doors and sloping bottoms.*—When the door is inclined the bottom is all of cast iron, and the aperture itself slopes so as to facilitate the discharge of the slices.

*Necessity for tight joints.*—Staunchness and water-tightness of all doors is secured by an indiarubber washer, against which screwing-up is done. In the wide bottom doors resort has to be made to what is known as the *hydraulic joint*, i.e., the indiarubber is

hollow, and, in tightening, water is injected into the interior under strong pressure, which presses the rubber very forcibly against the door.

*Line batteries versus circular batteries.*—The diffusers may be arranged in a straight line or in a circle. 1. Line batteries are the oldest form (see Fig. 22). They are generally arranged in two parallel rows, and the discharge doors are turned in the same direction to the inside of the double line, so that the exhausted slices fall between the two rows into a channel, in the bottom of which a helix turns and carries them away. The bottom of the channel is sometimes simply inclined with a sufficiently great slope to cause the pulp to run into a pit, from which it is extracted mechanically.

2. *Circular batteries.*—*Their many advantages.*—Circular batteries (Figs. 13, 21, 23) are very convenient because the cells are easily filled with beet-slices. They are very general in France. The exhausted pulp is discharged from the diffusers into a pit, in the centre of which an elevator or conveyor raises it to the pulp presses.

*Charging line batteries with beet slices.*—The charging of the line battery cell (Fig. 22) with sliced beets is done (1) by trucks which, filled underneath the root-slicers, roll on a short aerial railway, and empty themselves from below into the cell; or (2) by a gutta-percha belt running horizontally the whole of the length of the battery, at the bottom of a trough. The root-slicer drops the slices on the belt. In front of each cell the trough has a door (Fig. 22) opening so that the slices fall into the cell. The door, opening inwards, makes a barricade, and prevents the slices going farther on than the cell which is being filled.

*The method of charging circular batteries.*—Circular batteries (Figs. 13, 21 and 23) are charged differently. The root-cutter is on an upper floor. Under the root-cutter is a gigantic hopper or funnel, terminating in an inclined chute, which comes as far as the doors of the diffusers. The funnel rests on bearings so that it can turn on itself, and thus the end of the chute can be brought in front of each cell in turn. When the root-slicer is at work the slices automatically descend the chute and fall right into the cell, without need of any other method of transport.

*Calorisators, their description and function.*—Whether the battery be a line or a circular one, each cell carries its own calorisor, and the calorisor is a vertical bundle of pipes—surrounded by steam—in which the juice circulates as it passes from the one diffuser to the other (Fig. 23). At other times the calorisor is simply a very wide pipe containing a large steam coil. The calorisor is sometimes replaced, as in Belgium, by a simple injection of steam, which heats the juice in its passage through the pipe, and condenses there. This last arrangement is imposed by the Belgian Excise authorities, to avoid the leakage often incidental to other forms of calorisors,

which might injuriously affect the revenue, as the sugar [Excise tax in Belgium is levied on the quantity of juice produced. "

*The old-fashioned process of reheating the juice.*—Before caloristors were invented, the juice was reheated in two tanks fitted with

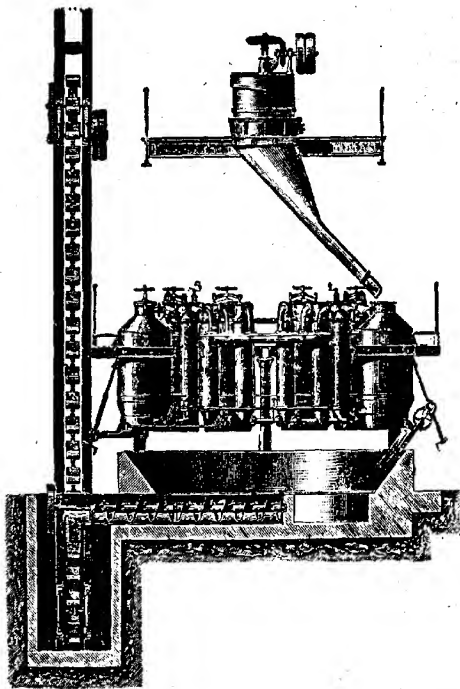


FIG. 23.—Circular battery, showing the way in which it is fed and the removal of the pulp by a helical conveyor (C.A.L.).

steam coils, or in large tubular reheaters, through which the juice passed from cell to cell. These two reheaters served for the whole battery, and involved two extra taps to each diffuser. This obsolete method is still sometimes met with.

*The valves all manipulated from the same stage.*—All the taps of

each cell are united in a single buffet, clearly and neatly arranged to facilitate operations. The keys of all the taps, or rather the wheels of all the valves, are thus on the same level, and within reach of the workman whether in front or behind the diffusers (Fig. 23).

*The working staff attached to a diffusion battery.*—In circular bat-



FIG. 24.—Beet diffuser. "Cell" of diffusion battery (CAIL).

teries and in line batteries with a conveyor the batteryman and an assistant are quite competent to do all the work required. But, when the cells are fed from trucks, three or four extra men are required to work them. Nevertheless, this last system is often preferred, as the diffusers are filled almost instantaneously, and much

*more work can be got out of the batteries—a great advantage where diffusion equipment is on a small scale.*

*Accumulations of air and vapours in the cells, and their removal.—*This is a frequent cause of trouble. The batterymen should frequently blow off these accumulations, but, working at a high temperature, they often neglect to reduce the pressure of steam on the calorisers at the right time. The juice thus becomes heated above boiling-point, and, when pressure is reduced, larger volumes of

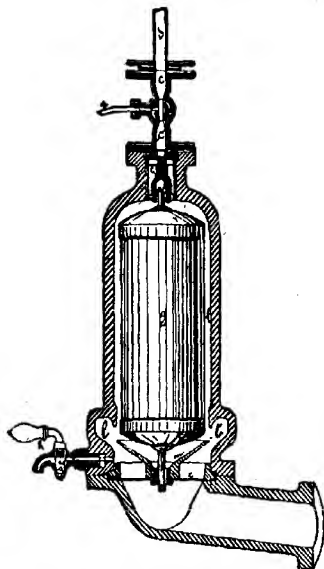


FIG. 25.—Apparatus for removing the accumulation of air and vapours in diffusion cells (K. LEYSER).

steam accumulate in the cells. This steam and the air in the cells are liable to "trap," and thus prevent the sugar being extracted in a regular and uniform manner from the slices. To meet this difficulty Leyser devised the appendage shown in Fig. 22. It consists of a float *g*, connecting by a spherical joint at its upper end with the valve *b*, and guided at its lower end by means of a rod at the centre of the strainer *e*. The tube *a* communicates with the diffuser. If vapours collect in the diffuser they will pass out through the tube *a*,

around the float *g*, through the valve *b* and tube *d* into the open air. Any foam that may collect in the cell will also escape. As soon as the juice in the cell rises to a sufficient height the float *g* will lift and close the valve and thus prevent the juice itself from escaping. The small funnel intercepts any fine pieces of pulp which may pass the strainer and prevents them from clogging the apparatus. The funnel is removed and emptied when needful. The valve between *cc* is closed when the apparatus is not in use.

*Plan and arrangement of diffusion batteries.*—*Single and double lines of batteries.*—Local conditions largely control the arrangement of a battery. A single line requires a very long building, but the easy removal of its exhausted slices and favourable conditions for enlarging plant are in its favour. The enormous length of the return pipes is objectionable. But the double line facilitates removal of exhausted slices; return pipes are very short, and manipulations are as simple as in the circular battery. In both single and double line batteries there is difficulty in charging the last cell in the series with slices without (1) either having slices left over, which fall on the floor, or (2) feeding this cell irregularly.

*Circular batteries.*—The circular battery involves a very lofty square edifice, and the methods available for removing the exhausted slices and the space occupied constitute its main drawbacks. But the actual cost of construction is very much the same in any case.

*Diffusion residuals—Exhausted pulp.*—*The excess of water must be removed from the water-logged pulp before it can become available as a cattle food.*—Exhausted pulp, as it comes from the cells, is very wet, owing (1) to the water with which it is distended; (2) to the physical phenomenon on which the process of diffusion is based, in virtue of which the cells of the exhausted slices are charged with water instead of the original saccharine juice.

*Had pulp presses been in vogue, diffusion would not have been abandoned at the outset.*—Such pulp is valueless for cattle-feeding purposes, the moisture injuriously affecting the health of the cattle fed on it. Hence the abandonment of Mathieu de Dombasle's process. If pulp presses had been then to the fore, however, no other system for extracting the juice would have henceforth prevailed.

*The many difficulties in the way of pressing the pulp.*—The pulp is difficult to press. It has been cooked in the diffusers at a temperature of 70° to 80° C. (158° to 176° F.) for at least half an hour; it has thus become soft and tacky by the effect of the heat on the pectic substances exposed by slicing. As all the cells under the slicing knife are naturally cut open and even often torn by blunt knives, that is one reason why the knives should be often sharpened.

*Its jelly-like nature a great drawback to its effectual pressure.*—When a fistful of pulp is pressed in the hand it is easily converted into a

jelly that strings between the fingers, especially when the diffusion has been heated a little too much so as to work a little more rapidly. The object should be to squeeze the pulp sufficiently without spoiling it. A roller press would simply triturate it without getting rid of the water. The machine, therefore, should slowly compress the pulp, and not bruise it at the time the greatest amount of pressure is being imparted.

*Kluzemann's press.*—The first press fulfilling the above requirements was Kluzemann's press, a vertical cast-iron cylinder resting on a cast-iron hood. In its axis there turns a piece having the form of a much elongated cone standing upright on its base, of the height of the cylinder, and the base of which is slightly smaller in diameter than the cylinder. This cone carries wide helical blades almost abutting on the cylinder, down to the bottom. If wet exhausted pulp be placed in the cylinder it is caught by the blades, forced to descend and drawn into the free space between the cone and the cylinder, a space which narrows more and more on account of the conical form of the central piece. The pulp is thus slowly pressed, its water escapes through the holes in the cylinder. When it reaches the bottom it meets a piece of cast iron which still further restricts the opening, and whose distance from the cylinder can be regulated so as to still further increase the pressure. The cylinder carries at intervals iron arms on the inside, which prevent the pulp from turning with the cone, and thus blocking or jamming it. The liquid which flows round the cylinder is collected in a gutter, and led into a drain.

*Bergneen's press.*—Kluzemann's press has been altered by Bergneen (Fig. 26). The conical kernel is also made to act as a filter, and the helix is in two parts, driven at different speeds. It is a little more powerful than Kluzemann's press. But the latter has also been improved so far that there is but little to choose between the two.

*Selwig and Lange's press.*—This press, constructed on a different principle, consists of two strong circular plates with a filtering surface fixed in a vertical frame. These plates turn slowly and in different directions with the same speed, only their axes are a little inclined, the one to the other, so that they approach each other very nearly at a certain point of their circumference. If pulp be introduced between the plates where they are the farthest apart, it is carried forward by the rotation, but as the pressure goes on increasing the pulp is caught and pressed, and finally escapes, sufficiently dried, after traversing half a circumference. This press—a fine machine—is extensively used. There are many other systems, but the three described produce the best marketable pulp.

*The conveyance of the pulp to the press.*—The pulp is lifted out of the pit of the diffusion battery by cup elevators (Figs. 21 and 23). The cups are perforated with numerous holes, sometimes even altogether open, to let the water, with which the pulp is mixed in the

bottom of the pit, escape. The chain of cups discharges into a horizontal trough with as many apertures as there are pulp presses

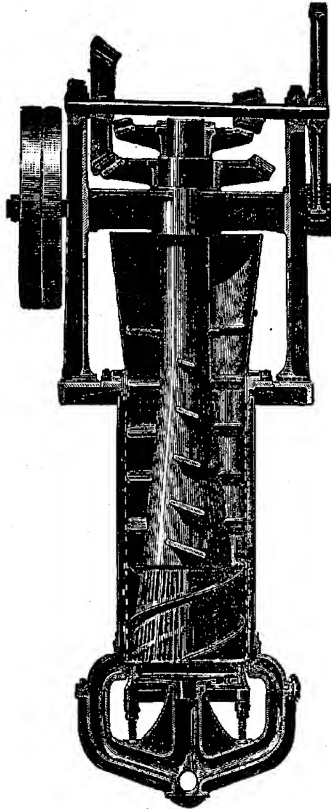


FIG. 26.—Bergneen's exhausted pulp press.

in the bottom of which a helix turns. These apertures open above the hopper of each press, and are closed when it is not working.

*The location of the pulp press floor.*—The pulp presses are gener-



ally high enough up for the dried pulp to pass into a room, below which the farmer's carts are loaded through a trap-door. Formerly the dried pulp was raised by a helical elevator, and discharged therefrom into the carts. The endless arrangements for lifting the pulp in different factories are dictated by circumstances and the plan of the factory.

*Siloing the pressed beet pulp.*—When there is no immediate sale for pulp it is siloed. These silos are long deep trenches, dug out of the ground and built on the sides with potter's clay, into which the pulp is run and covered with earth. The small amount of sugar, still left in the exhausted beet, first of all enters into *alcoholic* fermentation, which afterwards gives place to the *lactic* fermentation.

*The slight acidity of siloed pulp relished by cattle.*—The original shape of the fermented slices quite disappears, and they very soon only consist of a homogeneous mass, which cattle prefer to the fresh pulp—owing to the slight acid taste. It is not, however, given to cattle alone; it is mixed with cut straw or hay, and animals are then very fond of it. Pulp makes a valuable cattle food, says Wiley. Cattle may be fed therewith in the fresh state or after preservation in silos. Extensive experiments in drying pulp and preserving it in the dried state have been fairly successful. The value of the pulp for feeding purposes is from one-fourth to one-fifth of the value of the beets. Their feeding value may, however, be judged from the following analyses:—

TABLE XIII.—ANALYSES SHOWING NUTRITION VALUE OF BEET DIFFUSION PULP (RESIDUAL).

	Fresh. Per Cent.	Pressed. Per Cent.	Siloed. Per Cent.	Pressed with Lime. Per Cent.	Dried. Per Cent.	Maceration Residues. Per Cent.
Water . . .	94.0	89.8	88.5	65.0	11.6	78.9
Ash . . .	0.4	0.6	0.9	3.9	7.1	2.8
Albuminoids .	0.5	0.9	0.9	3.2	6.6	1.5
Cellulose . .	1.4	2.4	2.3	9.6	19.8	4.4
Carbohydrates.	3.6	6.1	7.2	17.8	54.8	12.3
Fat . . .	0.1	0.2	0.2	0.5	0.6	0.1

Spent pulp sells in France at from three to four shillings per ton. About three million tons are produced annually.

*Ratio of weight of exhausted pulp to original weight of beets submitted to diffusion.*—As the juice extracted by diffusion is replaced by an equal volume of water of unit density, the difference between the weight of the juice drawn from the "cells" and the weight of the water which replaces it will give the loss in weight which the beet slices undergo in the diffusion process. If 115 litres of juice of

density 1.05—corresponding to 13.3 kilogrammes of dissolved substances per hectolitre<sup>1</sup>—be extracted on an average from 100 kilogrammes of beets, it follows that  $115 \times 13.3 \div 100 = 15.295$  kilogrammes of dissolved matter have been extracted from the beet, say in round numbers, 15 per cent. Consequently 85 per cent. of pulp remains. The quantity of exhausted pulp therefore depends on the volume and the density of the diffusion juice, so that the co-efficient to be used is not always the same, and it must be determined each time stock is taken, and the factory books balanced. To avoid calculation, it has generally been agreed to accept the average figure of 85 per cent. During manufacture the spent pulp is analysed as often as possible. It is easily sampled. The spent slices as they come from the diffuser in normal working are uniformly exhausted, and all that has to be done is to take several handfuls and mix them into a convenient sample. The samples may also be taken from the elevator cups on their way to the pulp presses, taking a little from each cup. The mixed pulp is cut up, pressed and polarised. The sugar spent in the pulps may vary, but they are frequently analysed, and the daily average fairly represents the loss of sugar in this residual.

*The chemical control of the diffusion work of a beet-sugar factory during the night shift.*—There is some little difficulty in controlling chemically the night shift work. This is got over by utilising the very close connection between the sugar left in the spent pulp and that found in the drainage from the pulp presses, so that the analyses of the latter may replace that of the spent pulp to a certain extent. To get a fair average sample of the press liquor a known quantity of basic acetate of lead is run into a litre flask, and the night foreman runs a small sample of the drainage water into the bottle from time to time so that it is filled by about seven o'clock in the morning. The whole is then mixed, polarised, and the dilution due to basic acetate of lead taken into account. The easiest way is to add 100 c.c. of basic acetate solution and then run in about 50 c.c. of the liquor from the presses every half-hour, so that the litre is filled by morning. The liquid is filtered after mixing, polarised, and the sugar calculated for 100 c.c. of liquid, and the result multiplied by 1.1 so as to take the dilution into account. Calculation may be dispensed with by multiplying the degrees obtained by Laurent's saccharimeter with a tube 20 centimetres long by 0.18, but it would be better to use tubes 40 or 50 centimetres in length so as to get more accurate results. The coefficient will then be 0.09 and 0.072 instead of 0.18.

<sup>1</sup> Kilogrammes per hectolitre = lb. per 10 imperial gallons.

Litres per hectolitre = imperial gallons per 100 imperial gallons.

Grammes per hectolitre = parts by weight in 100,000 parts by volume, i.e., lb. per 10,000 gallons.

TABLE XIV.—ANALYSES OF WEAK LIQUORS (SCHEIBLER'S TABLE).

Density at 15° C. (59° F.).	Dissolved Matter.		Density at 15° C. (59° F.).	Dissolved Matter.	
	Per Cent. grammes.	Per Cent. c.c.		Per Cent. grammes.	Per Cent. c.c.
1.00000	0.00	0.0000	1.00198	0.51	0.5110
1.00004	0.01	0.0100	1.00202	0.52	0.5211
1.00008	0.02	0.0200	1.00206	0.53	0.5311
1.00012	0.03	0.0300	1.00210	0.54	0.5412
1.00016	0.04	0.0400	1.00214	0.55	0.5512
1.00020	0.05	0.0500	1.00218	0.56	0.5612
1.00023	0.06	0.0600	1.00222	0.57	0.5713
1.00027	0.07	0.0700	1.00226	0.58	0.5813
1.00031	0.08	0.0800	1.00230	0.59	0.5914
1.00035	0.09	0.0900	1.00234	0.60	0.6014
1.00039	0.10	0.1000	1.00237	0.61	0.6115
1.00043	0.11	0.1100	1.00241	0.62	0.6215
1.00047	0.12	0.1200	1.00245	0.63	0.6316
1.00051	0.13	0.1301	1.00249	0.64	0.6416
1.00055	0.14	0.1401	1.00253	0.65	0.6517
1.00059	0.15	0.1501	1.00257	0.66	0.6617
1.00062	0.16	0.1601	1.00261	0.67	0.6718
1.00066	0.17	0.1701	1.00265	0.68	0.6818
1.00070	0.18	0.1802	1.00269	0.69	0.6919
1.00074	0.19	0.1902	1.00273	0.70	0.7019
1.00078	0.20	0.2002	1.00276	0.71	0.7120
1.00082	0.21	0.2102	1.00280	0.72	0.7220
1.00086	0.22	0.2202	1.00284	0.73	0.7321
1.00090	0.23	0.2303	1.00288	0.74	0.7421
1.00093	0.24	0.2403	1.00292	0.75	0.7522
1.00097	0.25	0.2503	1.00296	0.76	0.7623
1.00101	0.26	0.2603	1.00300	0.77	0.7723
1.00105	0.27	0.2703	1.00304	0.78	0.7824
1.00109	0.28	0.2804	1.00308	0.79	0.7924
1.00113	0.29	0.2904	1.00312	0.80	0.8025
1.00117	0.30	0.3004	1.00316	0.81	0.8126
1.00121	0.31	0.3104	1.00319	0.82	0.8226
1.00125	0.32	0.3204	1.00323	0.83	0.8327
1.00128	0.33	0.3304	1.00327	0.84	0.8428
1.00132	0.34	0.3405	1.00331	0.85	0.8529
1.00136	0.35	0.3505	1.00335	0.86	0.8629
1.00140	0.36	0.3605	1.00339	0.87	0.8730
1.00144	0.37	0.3705	1.00343	0.88	0.8831
1.00148	0.38	0.3806	1.00347	0.89	0.8931
1.00152	0.39	0.3906	1.00351	0.90	0.9032
1.00156	0.40	0.4006	1.00355	0.91	0.9133
1.00160	0.41	0.4106	1.00359	0.92	0.9233
1.00163	0.42	0.4207	1.00362	0.93	0.9334
1.00167	0.43	0.4307	1.00366	0.94	0.9435
1.00171	0.44	0.4408	1.00370	0.95	0.9536
1.00175	0.45	0.4508	1.00374	0.96	0.9636
1.00179	0.46	0.4603	1.00378	0.97	0.9737
1.00183	0.47	0.4709	1.00382	0.98	0.9838
1.00187	0.48	0.4809	1.00386	0.99	0.9938
1.00191	0.49	0.4910	1.00390	1.00	1.0039
1.00195	0.50	0.5010			

TABLE XV.—FOR THE ANALYSES OF WEAK SUGAR LIQUORS.  
(French saccharimeter. Normal weight, 16.29 grammes.)

Sacchari- metrical Degrees.	Sugar per 100 c.c.		Sacchari- metrical Degrees.	Sugar per 100 c.c.	
	400-mm. tube.	500-mm. tube.		400-mm. tube.	500-mm. tube.
0.1	0.0081	0.0065	3.1	0.251	0.201
0.2	0.0163	0.0130	3.2	0.259	0.208
0.3	0.0243	0.0195	3.3	0.267	0.214
0.4	0.0326	0.0260	3.4	0.275	0.221
0.5	0.0405	0.0325	3.5	0.283	0.227
0.6	0.0486	0.0390	3.6	0.291	0.234
0.7	0.0567	0.0455	3.7	0.300	0.240
0.8	0.0648	0.0520	3.8	0.308	0.246
0.9	0.0729	0.0585	3.9	0.316	0.253
1.0	0.081	0.065	4.0	0.325	0.260
1.1	0.089	0.071	4.1	0.332	0.266
1.2	0.097	0.074	4.2	0.340	0.273
1.3	0.105	0.084	4.3	0.348	0.279
1.4	0.113	0.091	4.4	0.356	0.285
1.5	0.121	0.098	4.5	0.364	0.292
1.6	0.129	0.104	4.6	0.373	0.299
1.7	0.138	0.110	4.7	0.381	0.305
1.8	0.146	0.117	4.8	0.389	0.312
1.9	0.154	0.124	4.9	0.397	0.318
2.0	0.163	0.130	5.0	0.405	0.325
2.1	0.170	0.137	5.1	0.413	0.331
2.2	0.178	0.143	5.2	0.421	0.338
2.3	0.186	0.150	5.3	0.429	0.344
2.4	0.194	0.156	5.4	0.437	0.351
2.5	0.202	0.162	5.5	0.445	0.357
2.6	0.210	0.169	5.6	0.453	0.364
2.7	0.218	0.175	5.7	0.461	0.371
2.8	0.227	0.182	5.8	0.469	0.377
2.9	0.235	0.188	5.9	0.477	0.383
3.0	0.243	0.195	6.0	0.486	0.390

The spent slice is a very watery body of a gluey nature, from which it is difficult to press all the liquid out of it. The analysis of spent beet slices is thus somewhat complicated. A different method of analysis has to be adopted to that for beets. The slice is divided by a mince-meat knife into small fragments as minutely as practicable, and a known weight of the substance is weighed out into a basin, water added and heat applied. The sugar diffuses into the water. The liquid is run off into a graduated flask, the pulp again washed with boiling water, likewise run into the flask, when the pulp is sufficiently exhausted. As the pulp does not contain

much sugar, the error is trifling. The liquid is defecated, made up to the mark, filtered and polarised in a 400 or 500 millimetre tube. A rule of three sum gives the percentage of sugar in the pulp. It is better, as in Pellet's method (p. 14), to introduce the pulp itself into the flask, heat with water and deduct the volume due to the woody fibre. The loss of sugar may be calculated from the weight of the spent pulp and that of the wash water, if they be known, and their analysis. Such calculations are, of course, mere estimations; but it is immaterial. If the analysis gives the lowest possible results that is all that is necessary. All we want to know is whether the spent pulp contains more or less sugar, so that the batteryman may work his battery so as to prevent as little loss as possible, or at least limits himself to a figure agreed upon between himself and the works manager, who should find out whether the cost of extracting the sugar remaining in the pulp does not cost more than the value of the sugar itself. A good manager endeavours to extract as much sugar from the beet as possible, but he should not go too far, as the cost then exceeds the gain. It all depends on the attention paid to these matters whether the manager is a good or bad administrator of his factory.

TABLE XVI.—EXHAUSTED PULP.

(Juice of exhausted pulp. 400-millimetre tube. Normal weight of instrument, 16·2 grammes; addition of  $\frac{1}{4}$  basic acetate of lead.)

Sacchari- metrical Degrees.	Sugar Per Cent.	Sacchari- metrical Degrees.	Sugar Per Cent.	Tenths.	
				Degrees.	Sugar Per Cent.
1	0·09	10	0·89	0·1	0·01
2	0·18	11	0·98	0·2	0·02
3	0·27	12	1·07	0·3	0·03
4	0·36	13	1·16	0·4	0·04
5	0·45	14	1·25	0·5	0·05
6	0·53	15	1·34	0·6	0·06
7	0·62			0·7	0·07
8	0·71			0·8	0·08
9	0·80			0·9	0·09

# DIFFUSION

65

TABLE XVII.—EXHAUSTED PULP.

(Table for German instruments. Juice of exhausted pulp. 400-millimetre tube. Normal weight of instrument, 26·048 grammes; addition of  $\frac{1}{16}$  basic acetate of lead.)

Sacchari- metrical Degrees.	Sugar Per Cent.	Sacchari- metrical Degrees.	Sugar Per Cent.	Tenths.	
				Degrees.	Sugar Per Cent.
1	0·14	10	1·43	0·1	0·02
2	0·29	11	1·57	0·2	0·03
3	0·43	12	1·72	0·3	0·04
4	0·57	13	1·86	0·4	0·05
5	0·72	14	2·00	0·5	0·07
6	0·86	15	2·14	0·6	0·08
7	1·00			0·7	0·10
8	1·14			0·8	0·11
9	1·29			0·9	0·15

*Pfeiffer's automatic discharge diffusion process.*—By this process the following plant items may be dispensed with, viz. : (1) the lower doors of the cells; (2) the pulp helix; (3) the pit; (4) the pulp elevator; and (5) the wash-liquor pump. In "cells" with bottom discharge (Fig. 24), the bottom door is about equal in diameter to that of the body of the cell. It is best to fix a rather narrow limit, so as not to exceed a maximum height of beet slices, whatever may be the capacity of the cell, and obtain on drawing off from below a liquor of high density, easy and uniform circulation, and the most satisfactory exhaustion. The central entrance and exit of the juice facilitate these results, but the juice, entering by the centre of the upper door, passes preferably through the beet slice nearest its entrance, and that at the expense of the slices farther away alongside the periphery. Analogous phenomena occur at the exit of the diffuser; all the circulation channels converge towards the exit orifice, in the centre, so that the best exhausted portion in the mass of the diffuser assumes an oval form. That should therefore be the form of the theoretical diffuser. This natural circulation is avoided by increasing the surface perforation of the centre towards the periphery, so as to ensure a uniform entrance of the juice on the plane of the beet slices, limited by the perforated wrought iron plates, but the calculation of the best perforations to adopt is based on approximations without exact data. The conical form of the fixed bottom of the cells, adopted for automatic discharge in Pfeiffer's patent (Fig. 27), the slope of which is the practical result of two years' trials and two years' actual working, has therefore many

advantages. It is lined with perforated wrought iron; a discharge orifice placed at the lower part of the cone, and therefore in the centre, induces the circulation as in the ordinary diffuser. On this

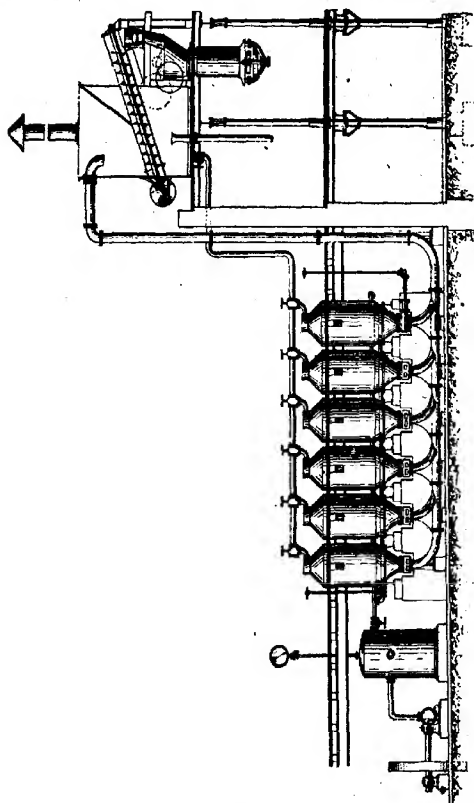


FIG. 27.—Pfeiffer's automatic discharge diffusion process (Can).

double bottom is a crown, closed by a valve, manipulated from the upper floor, through which air enters during the discharge. A tight valve, with a hydraulic joint, also wrought from above, closes the

beak of the cone, which connects farther on with the cast-iron piping passing through the axis, and below the cells, and rising again directly to open in the discharge tank placed above the pulp presses. This tank is divided into two separate superimposed parts, the upper part retains the pulp, which is distributed regularly to the presses; the double bottom collects the wash waters, the overflow of which runs on to the washers, and the portion kept in reserve below the level, regulated by the overflow, is used before each discharge to fill the discharge pipe, partially emptied by the preceding operation. A compressor, the capacity and the force of pressure of which are determined according to size of battery and height of presses, forces a suitable volume into a recipient. A manometer placed on the recipient, and brought well within sight of the battery, indicates the pressure of the air, say 2 kilogrammes per sq. cm.; as soon as this pressure is reached an arrangement, acting on the distribution of the compressor, enables the latter to discharge its functions without absorption of work. In discharging, the cell being isolated, the lower valve is opened, and also the wash-liquor valve, to fill the pipe. The latter is closed almost at once, and the compressed air turned on. In all, three valves are manipulated from the upper floor; the operator watches his manometer, the pressure of which rapidly descends to 0, which then indicates that air is passing through the pipe into the discharge tank. The operation, which lasts 30 seconds in the case of a diffuser of 50 hectolitres (1,100 gallons), is finished, the compressed air is turned off, the discharge valve closed, and the upper door is opened for the refilling of the cell with fresh beet slices. The operation is very simple, and the battery may thus be laid down on very slight masonry, the pit being suppressed. The greatest cleanliness is ensured below the stage of the diffusers, where no trace of water, juice, or pulp is to be seen; the operation goes on regularly and without breakdowns.

*Pellhardt's eliminator of pulp from diffusion juice.*—This machine for eliminating pulp from diffusion juice retains stray pulp and the pieces of pulp carried with the juice into the measuring vessels. It frees the juice from all solid substances, in suspension, which increase the volume and weight shown by the hydrometer. It increases the purity of the juice, facilitates the working of the carbonatation process, and prevents the conveyance of the pulp into the reheaters, carbonators, etc., where it accumulates. It is claimed that this is the only machine which is really practical. It works without interrupting the circulation of the juice in the diffusion battery, and does not require cleaning. That is why it is the only one authorised by the Belgian Excise, where the tax is levied on the diffusion juice. It consists (Fig. 28) of a cylinder ending in a small cone, of a peculiarly shaped perforated mobile cylinder, forming a scraper and a



flap valve for discharging. It is driven by cog-wheels and pulleys. The juice from the battery runs into the machine through a pipe half-way up the sieve, and passes into the interior of the perforated cylinder. Thence it flows through an elbow into the pipe leading into the measuring vessels. The pulp drawn over with the juice is retained by the revolving perforated cylinder and accumulates in the conical part, from which it is discharged by the manipulation of a valve in one of the diffusers of the battery. The machine is not emptied except when the diffuser in question is half-filled with fresh beet slices. This perforated cylinder revolves at a speed of 10 to 12 revolutions a minute. A scraping arrangement continually removes the pulp from the cylinder. The machine may be fixed on the stage of the diffusion battery between the juice pipe from the battery to the measuring tanks. The discharge pipe should be

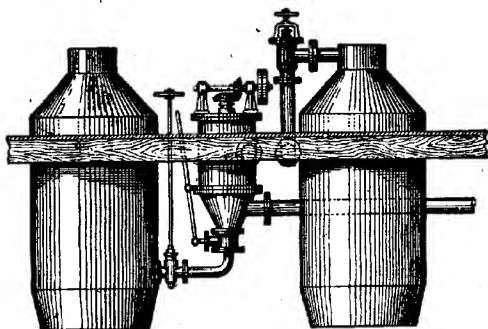


FIG. 28.—Showing Pellhardt's pump eliminator fixed between two diffusers (MAGUIN).

at a third of the height of the diffuser with which it is connected, should have an internal diameter of 70 to 80 millimetres ( $2\frac{1}{2}$  to 3 inches), and be closed by a valve or tap fixed to the body of the diffuser. The flap valve is wrought by a rod worked from the diffusion stage. The working of the machine requires little supervision. It is not opened to remove the deposit. There is thus no interruption in the circulation. It is discharged by opening the tap, fixed to the cell, when the latter is almost half-filled with fresh beet slices. Then the flap valve is opened and the deposit flows into the diffuser. When the machine is empty the valve is first closed, then the tap. The inside scraper should touch the perforated cylinder. It is therefore drawn near to it by two outside screws, fixed in the outside cylinder of the machine. The small air tap fixed to the lid is opened to let the air escape.

### CHAPTER III.

#### CARBONATATION AND FILTRATION.

*THE properties and nature of the juice.*—Crystallisation or concentration prevented by gelatinisation of pectic substances.—Beet juice is a more or less deep violet-coloured turbid liquid, with a fresh smell when extracted from sound beets. The raw juice cannot be evaporated, as it contains much pectic, albuminoid, and other impurities, which transform it when heated into a gelatinous confection or jelly, from which it would be well-nigh impossible to extract the smallest amount of sugar. The term jelly is used advisedly, because it is to the pectic matters of fruits that these sweetmeats owe their gelatinous form.

*Defecation.*—Elimination of the pectic substances from the juice by lime.—The manufacture of sugar from sugar-cane juice being much older than the extraction of sugar from beet juice, it was quite natural that the process used in the purification of beet juice should be copied from the more ancient sister industry. The purification of cane juice, termed defecation, was effected by simply boiling the juice with lime. The same process was extended to beet juice.

*The action of lime on the juice of the sugar beet.*—When a small quantity of lime, say a few thousandth parts, is incorporated with beet juice, the lime coagulates certain principles of the juice, and this coagulum eventually falls to the bottom of the vessel, whilst the supernatant juice is converted into a straw-yellow and perfectly limpid liquid. But this process is a tedious and prolonged one, requiring a certain amount of time before it operates effectually. However, by boiling the lime-treated juice a thick layer of scum floats on the surface, whilst a rather heavy coagulum is precipitated to the bottom of the vessel, and the intervening liquid becomes perfectly limpid in a few minutes. The juice is defecated. In actual practice jacketed pans were used, into which the juice and lime were run, and the whole brought to the boil, the scum skimmed off, and then, when precipitation was complete, the clear limpid liquid was run off for further treatment. The liquid from the jacketed defecation pans was evaporated, then boiled over a naked fire until it assumed a syrupy condition, when it was run into crystallising basins

or moulds, where it was left to drain, and finally clarified or refined, to free it from molasses. Defecation was a long process. Many attempts were made by chemists to improve it, the more so as it was so liable to failure and mishaps.

1792. *Achard's defecation process*.—Achard got good results in defecating beet juice by *sulphuric acid* instead of lime, but it was a dangerous process, as a small trace too much acid completely destroyed the sugar.

1825. Mathieu de Dombasle, then Chaptal, and lastly Dubrunfaut, used *sulphuric acid in conjunction with lime*, so as to saturate or neutralise the excess of lime without rendering the juice acid.

1849. *Rousseau's carbonation process*.—The above very successful process lasted until about 1849, when it was superseded by Rousseau's.

*Description*.—Rousseau's process consisted in treating the limed juice, after decantation, by carbonic acid gas, from a charcoal furnace, until the excess of lime was saturated. This idea, first suggested by Kuhlmann in 1833, taken up by Barruel, and brought to a successful issue by Rousseau, was rapidly adopted in sugar factories. It was more economical than Dubrunfaut's, as it effected a great saving in bone char, the carbonate of lime being much less soluble in sugar solutions than the sulphate.

TABLE XVIII.—SOLUBILITY IN SUGAR SOLUTIONS OF CERTAIN INSOLUBLE SUBSTANCES (JACOBSTHAL).

Solubility in a Sugar Solution of—	5 Per Cent.	10 Per Cent.	15 Per Cent.	20 Per Cent.	25 Per Cent.
Calcium sulphate . .	2.095	1.946	1.593	1.539	1.333
„ carbonate . .	0.027	0.036	0.024	0.022	0.008
„ oxalate . .	0.033	0.047	0.012	0.068	0.001
„ phosphate . .	0.029	0.028	0.014	0.018	0.005
„ citrate . .	1.813	1.578	1.505	1.454	1.454
Magnesium carbonate .	0.317	0.199	0.194	0.213	0.284

1859. *Perrier and Possoz's double carbonation process*.—*Jelinek's process*.—Perrier and Possoz, in 1859, in conjunction with the Cail Engineering Company, brought out the double carbonation process, now adopted by all beet-sugar factories throughout the world. In Austria this system, almost without modification, is known as the Jelinek process; it is also termed the turbid defecation process; but the process, however known, involves the same method of working, which has, nevertheless, been slightly varied from time to time.

*The chemical composition of beet juice*.—To understand the chemical

reactions and phenomena involved in *defecation*, it is necessary first of all to know the composition of the sugar beet and that of its juice; the detailed chemical composition of both of which is given below :—

TABLE XIX.—COMPOSITION OF THE SUGAR BEET AND ITS JUICE.<sup>1</sup>

Water, 79 to 84.5 per cent.	} Juice.
Dry matter, 15.5 to 21.0 per cent., of which—	
Soluble in water, 11.5 to 17.0 per cent.	
Insoluble in water, 4 to 5 per cent.	
Ingredients of juice—	
I. Water . . . . . 80 per cent.	
II. Dry matter . . . . . 20 „	
A. Sugar . . . . . 15 per cent.	
B. Non-saccharine matter . . . . . 5 „	
(a) Ash, 0.8.	
(1) Incombustible salts—	
Potassium, sodium, rubidium, vanadium, calcium, magnesium, iron and manganese, combined with chlorine, sulphuric, phosphoric, silicic and nitric acids.	
(2) Salts transformed by combustion into carbonates. The same metals combined with oxalic, citric, malic and succinic acids.	
(b) Nitrogenous matter, 1.60 per cent.	
Proteins (albumen, etc.).	
Plasmatic substances.	
Asparagin ( $C_4H_7N_2O_3$ ), and various amides.	
Betaine ( $C_5H_{11}N_2O_3 + H_2O$ ).	
Glutamine ( $C_5H_9(NH_2)(CONH_2)CO_2H$ ).	
Leucine ( $NH_2C_6H_{10}CO_2H$ ).	
Tyrosine ( $C_9H_9(OH)(C_2H_5(NH_2)CO_2H$ ).	
(c) Non-nitrogenous bodies, 1.0.	
Raffinose ( $C_{18}H_{32}O_{16} + 5 H_2O$ ).	
Arabinose ( $C_5H_{10}O_5$ ) <sup>n</sup> .	
Dextrine ( $C_6H_{10}O_5$ ) <sup>n</sup> .	
Soluble pectic substances.	
Chlorophyll.	
Chromogene.	
Fat.	
Coniferin ( $C_{16}H_{22}O_8 + 2 H_2O$ ).	
(Vanillin, $C_8H_8O_2$ ).	
(d) Cellulose bodies, 1 per cent.; pectic, 0.6 per cent., and colouring substances.	
(e) Cholesterin ( $C_{26}H_{44}OH + Ag$ ).	

*The analysis of weighed versus measured quantities.*—Although the analytical processes for beet juice are uniform in all countries, yet the results are not expressed identically. In Germany all results

<sup>1</sup> The composition of sugar-cane juice is given on p. 236, and it may be useful for those interested to compare the two.

are brought to 100 parts by weight, *i.e.*, grammes per 100 grammes of juice. But in France the analytical results are expressed in kilogrammes per 100 litres of juice, *i.e.*, per 100 parts by measure (say lb. per 10 gallons). The German results are simply the French results divided by the density. It is usually more rational to bring the figures to the volume of the juice, as that is the only factor known, and is deduced from the capacity of the containing vessels. But where temperature comes into play the percentage by weight is the most rational course to follow in expressing the results. The weight of the juice per 100 kilogrammes of beet should therefore be determined; the weight is a factor which does not vary with the temperature. In beet analysis by the indirect method the sugar per cent. of juice ought always to be brought to 100 parts by weight of grammes of juice, and not to 100 c.c., the volume of juice having no constant ratio with the weight of the beets.

*Determining the density of juice.*—*The use of hydrometers.*—The density may be taken by Mohr's hydrostatic balance, an accurately graduated, as well as sensitive, hydrometer, or by the special areometers of Brix or Balling graduated into tenths of a degree.

*Influence of temperature.*—It is necessary to work at the temperature at which the instrument is graduated, a condition often difficult to realise; use is therefore made of correction tables (see Table V., p. 9). The degrees Brix show the apparent percentage of dissolved substances.

The actual percentage of sugar is determined by direct polarisation if the juice be clear enough, otherwise it is treated with a 10 per cent. solution of basic acetate of lead and filtered. The results are either multiplied by 1.1, or the observation made in a 220 millimetre tube. The polarimetric degrees are multiplied by 1.629 for the French saccharimeter, and 2.6048 for the German saccharimeter, and the result then expressed in grammes (*a*) per 100 c.c., or (*b*) per 100 grammes of juice. All calculations may be dispensed with by using a flask graduated in three different places, namely, 100, 110, and 128 c.c. 100 c.c. of juice are drawn off, basic acetate of lead added to about 110, or less, as occasion may require, and then brought up to the mark of 122.8 c.c. The filtrate contains exactly the normal French weight, 16.29 grammes in 100 c.c., so that by dividing by 5, or multiplying by 2 the percentage of sugar is obtained. When a beet is pulped and the juice pressed, the juice contains, besides sugar and water, soluble mineral salts, soluble albumen, and other organic bodies, such as certain acids, asparagin, etc.

*The pectic and pectose fermentation of the unpurified juice.*—If the crude juice be allowed to stand for some time, the pectic matters ferment and are transformed into two gelatinous acids—the pectic and the pectosic. The juice then segregates into a jelly, or, if

diluted with water, it strings, like certain white wines, after they have gone wrong.

*Secondary fermentations.*—The juice, therefore, cannot be kept for any length of time, as secondary fermentations set in, which change the sugar into uncrystallisable glucose or mannite.

*Beneficial effect of liming the juice.*—If lime be incorporated with cold juice, the organic acids are neutralised, and the pectic bodies changed into pectate of lime, which falls to the bottom of the vessel. The liquid may then be preserved indefinitely if the lime be not in excess. Maumené suggested this method for preserving and storing the juice and extracting the sugar from it all the year round.

*Bad effect of an excessive application of lime.*—*Production of lactic and butyric acid fermentation.*—But, if lime be used in very great excess, a special fermentation, the *lactic*, due to the albuminoids, is developed, followed by the *butyric* fermentation, the latter giving off a most disgusting odour, the cause of the bad smell of the recesses of badly kept sugar factories.

*Defecation.*—*The rationale of the process.*—When crude juice is boiled without lime the albumen is coagulated; but not the pectic matters if ebullition be not carried too far. But when the juice is boiled with lime both the albuminous and the pectic impurities are precipitated, as well as certain organic lime salts. This constitutes the *Defecation Process*, properly so called.

Should the lime not be used in too great excess, the dose being calculated so as to precipitate the organic impurities alone, a clear limpid juice is obtained. But the juice is turbid if the lime be used in excess. The cause of the turbidity of hot juices, when treated with an excess of lime, is due to the action of the lime on the sugar. The sugar, in fact, combines with the lime to form *sucrate* of lime. Now there are several compounds of sugar with lime, according to the number of equivalents of lime combined with one equivalent of sugar. *Monobasic*, *dibasic*, and *tribasic* sucrares of lime are known. *Monobasic* salts containing one equivalent of base are known as *normal* salts, whilst those with more than one equivalent of base are termed *basic* salts, e.g., basic acetate of lead.

*Solubility of the monobasic, dibasic, and tribasic sucrares of lime.*—Both the *monobasic* and *dibasic* sucrares of lime are soluble in water, but the *tribasic* sucrate of lime is insoluble. When a solution of a soluble sucrate is boiled it becomes turbid, by the formation of *tribasic* sucrate. From numerous experiments made by Horsin-Deon it would appear that the effect of heat is to cause the lime to combine with the least possible amount of sugar, and, as the sucrate containing the least amount of sugar is the *tribasic* sucrate, it is this sucrate which is formed and precipitated.

*The formation of the tribasic sucrate to be avoided.*—Consequently,

if a juice thus limed be filtered whilst boiling, a portion of the sugar will remain on the filter in the form of sucrate. The formation of the *tribasic* sucrate has to be guarded against, so as not to lose sugar in the sludge. It must also be borne in mind that the *tribasic* sucrate redissolves in the excess of sugar when the juice cools.

*All sucrares of lime unstable compounds.*—Sucrate of lime, in whatever state of combination it exists, is a very unstable combination. The least acidity decomposes it in all cases; sulphuric acid, carbonic acid, and alkaline carbonates easily decompose it, with formation of the corresponding lime salt.

*The practical application of the decomposition of sucrares of lime by  $\text{CO}_2$ .*—By injecting carbonic acid gas into a juice rendered turbid by sucrate of lime, insoluble carbonate of lime is precipitated and sugar liberated. It is the inherent principle involved in this reaction which has made Rousseau's process such a success, because there is no longer any need to fear excess of lime in the juice, carbonic acid being ready on the spot to neutralise it and thus prevent any possible loss of sugar from such excess.

*The sucrocarbonates and the risk involved in their formation.*—But there is still a danger in Rousseau's process—the solubility of carbonate of lime in sugar, and the formation of an insoluble sucrocarbonate which, passing into the sludge, involves a loss of sugar.

*Boivin's and Loiseau's researches on the sucrocarbonates.*—Boivin and Loiseau, about the year 1868, examined a compound of sugar, lime and carbonic acid, which they termed the hydro-sucrocarbonate of lime ( $6\text{CaO}, \text{C}_{12}\text{H}_{22}\text{O}_{11}, 3\text{CO}_2$ ) in which one equivalent of sugar was found in combination with six equivalents of lime and three of carbonic acid. This substance forms a jelly in the midst of sucrocalcareous solutions, which, if they are limpid, become turbid after having absorbed a certain amount of carbonic acid gas. But an excess of carbonic acid gas decomposes them. These facts have been observed, without any attempt at explanation, so far back as 1825, by Dubrunfaut. The subject was again taken up later on by Feltz, simultaneously with Boivin and Loiseau. Horsin-Deon in 1871 exhaustively examined the whole question. The combined result of all these researches was to demonstrate that, as soon as a certain amount of carbonic acid gas is injected into a limed juice, a certain amount of *hydro-sucrocarbonate* of lime is precipitated, and that it is indispensable to continue the carbonatation to destroy it, and that, if the operation be not pushed far enough, a certain portion of sugar is lost in the sludge. To avoid all loss of sugar in the form of sucrocarbonates the right point of saturation must be ascertained, as has been very well done since then by the help of the laboratory. But at the time of Rousseau's process chemistry found little favour in sugar factories, so, working without a guide, the manufacturer was exposed to unavoidable mishaps.

*Summary.*—The defecation of the juice is effected by the consecutive reactions which occur when it is treated first with lime and then with carbonic acid. The sucrate of lime formed liberates the sugar from the organic substances, and sets them free to combine with the lime in excess, the sugar having now lost its solvent action on them. The carbonate of lime formed acts, like the base of a coloured lake, on certain soluble impurities, *e.g.*, the colouring principles, which are “struck” on to the lime, hence the dark colour of the sludge. The sucrocarbonate formed during these lateral reactions is destroyed in its turn, and the last reactions of the nascent carbonate of lime completes the purification of the juice.

*Failure of all attempts to supersede lime in the purification of the juice.*—The several compounds which sugar forms with lime, and which are afterwards decomposed so as to liberate the sugar, when the purification of the sugar is complete, are highly interesting, and to them are due the success of the use of lime in the purification of the juice. Notwithstanding many attempts to find substances which might advantageously replace lime and carbonic acid, these two still remain the only purifying reagents in general use.

TABLE XX.—AMOUNT OF LIME ABSORBED BY SYRUPS OF DIFFERENT DENSITIES WITH DENSITIES OF RESULTING SYRUPS AND THEIR PERCENTAGE COMPOSITION (FELIGOT).

Quantity of Sugar Dissolved in 100 parts of Water.	Density of Syrup.	Density of Syrup when Saturated with Lime.	100 parts of Residue Dried at 120° C. contain—	
			Lime.	Sugar.
40.0	1.122	1.179	21.0	79.0
37.5	1.116	1.175	20.8	79.2
35.0	1.110	1.165	20.5	79.5
32.5	1.103	1.159	20.3	79.7
30.0	1.096	1.148	20.1	79.9
27.5	1.089	1.139	19.9	80.1
25.0	1.082	1.128	19.8	80.2
22.5	1.075	1.118	19.3	80.7
20.0	1.068	1.104	18.8	81.2
17.5	1.060	1.092	18.7	81.3
15.0	1.052	1.080	18.5	81.5
12.5	1.044	1.067	18.3	81.7
10.0	1.036	1.053	18.1	81.9
7.5	1.027	1.040	16.0	83.1
5.0	1.018	1.026	15.3	84.7
2.5	1.009	1.014	13.8	86.2



TABLE XXI.—PERCENTAGE OF QUICKLIME (CaO) IN MILK OF LIME OF DIFFERENT DENSITIES (MATEGZEK).

Degrees Baumé.	Per Cent. by Weight of CaO.	100 Litres		Degrees Baumé.	Per Cent. by Weight of CaO.	100 Litres	
		Weigh in Kg. (See n. 1.)	Contain Kg CaO. (See n. 2.)			Weigh in Kg. (See n. 1.)	Contain Kg CaO. (See n. 2.)
10	10.60	125.9	13.3	38	19.72	149.8	29.5
11	11.12	127.4	14.2	39	19.80	149.9	29.6
12	11.65	129.2	15.2	40	19.88	149.9	29.8
13	12.18	130.8	16.1	41	19.95	150.0	29.9
14	12.68	132.6	17.0	42	20.03	150.0	30.1
15	13.20	134.5	18.0	43	20.10	150.0	30.2
16	13.72	136.3	18.9	44	20.16	150.1	30.3
17	14.25	138.2	19.8	45	20.22	150.1	30.4
18	14.77	139.9	20.7	46	20.27	150.1	30.5
19	15.28	141.7	21.6	47	20.32	150.2	30.6
20	15.68	143.6	22.4	48	20.37	150.2	30.7
21	16.10	145.1	23.3	49	20.43	150.3	30.7
22	16.52	146.2	24.0	50	20.48	150.3	30.8
23	16.90	146.9	24.7	51	20.53	150.3	30.9
24	17.23	147.4	25.3	52	20.57	150.4	31.0
25	17.52	147.8	25.8	53	20.62	150.4	31.1
26	17.78	148.1	26.3	54	20.66	150.4	31.1
27	18.04	148.4	26.7	55	20.70	150.5	31.2
28	18.26	148.6	27.0	56	20.74	150.5	31.3
29	18.46	148.8	27.4	57	20.78	150.5	31.3
30	18.67	149.0	27.7	58	20.82	150.5	31.4
31	18.86	149.1	27.9	59	20.85	150.6	31.4
32	19.02	149.2	28.2	60	20.89	150.6	31.5
33	19.17	149.3	28.4	61	20.93	150.6	31.5
34	19.31	149.4	28.7	62	20.97	150.6	31.6
35	19.43	149.5	28.9	63	21.00	150.6	31.6
36	19.53	149.6	29.1	64	21.03	150.7	31.7
37	19.63	149.7	29.3	65	21.05	150.7	31.7

*Boutron and Boudet's soap test.*—White Marseilles soap. Dry 100 grammes alcohol 1600 c.c. Boil, filter, add filtrate to 1000 c.c. distilled water. Standardise with Ba Cl<sub>2</sub> (0.55 gramme in a litre of distilled water), 40 c.c. = 22 French degrees, 1° French = 0.56° German, 0.70 British. 1° French soap test =

Quicklime	0.0057	Magnesium sulphate	0.0125
Calcium chloride	0.0014	Sodium chloride	0.0120
Calcium carbonate	0.0103	„ sulphate	0.0146
„ sulphate	0.0140	Sulphuric acid	0.0082
Magnesia	0.0042	Chlorine	0.0075
Magnesium chloride	0.0030	Soap 50 per cent. aq.	0.1061
„ carbonate	0.0088	Carbonic acid	5 c.c.

<sup>1</sup> Or 10 gallons weigh in lb.

<sup>2</sup> Or 10 gallons contain CaO in lb.

# CARBONATATION AND FILTRATION

77

TABLE XXII.—TABLE GIVING THE QUANTITY OF MILK OF LIME TO ADD PER HECTOLITRE OF JUICE EXTRACTED (UNGER).

(By means of this table the quantity of quicklime to be added in the form of milk of lime may easily be ascertained.)

Quicklime Per Cent. to be Added.	Degrees Baumé.										
	10	11	12	13	14	15	16	17	18	19	20
	Litres.										
2.00	21.266	19.2307	17.3913	15.6790	14.5965	13.6135	12.5786	11.7647	11.0497	10.3627	9.7087
2.10	22.3404	20.1923	18.2509	16.6667	15.3285	14.1892	13.2075	12.3529	11.6022	10.8808	10.1942
2.20	23.4043	21.1538	19.1305	17.4603	16.0584	14.8849	13.8365	12.9412	12.1547	11.3990	10.6796
2.30	24.4681	22.1154	20.0000	18.2540	16.7883	15.5405	14.4554	13.5294	12.7072	11.9171	11.1650
2.40	25.5319	23.0789	20.8696	19.0476	17.5182	16.2162	15.0943	14.1176	13.2597	12.4352	11.6505
2.50	26.5957	24.0385	21.7392	19.8413	18.2482	16.8918	15.7232	14.7058	13.8122	12.9633	12.1359
2.60	27.6595	25.0000	22.6088	20.6349	19.0781	17.6474	16.3521	15.2841	14.3646	13.4710	12.6213
2.70	28.7233	25.9611	23.4784	21.4286	19.7080	18.2490	16.9810	15.8528	14.9171	13.9895	13.1068
2.80	29.7871	26.9231	24.3480	22.2222	20.4379	18.9189	17.6100	16.4706	15.4696	14.5077	13.5922
2.90	30.8509	27.8847	25.2174	23.0159	21.1678	19.5945	18.2389	17.0588	16.0221	15.0269	14.0777
3.00	31.9149	28.8463	26.0870	23.8035	21.8978	20.2701	18.8678	17.6471	16.5746	15.5439	14.5631
3.10	32.9787	29.8078	26.9566	24.6032	22.6277	20.9458	19.4967	18.2353	17.1271	16.0620	15.0485
3.20	34.0426	30.7694	27.8262	25.3968	23.3576	21.6214	20.1266	18.8235	17.6796	16.5802	15.5340
3.30	35.1064	31.7308	28.6958	26.1904	24.0876	22.2970	20.7547	19.4117	18.2921	17.0894	16.0194
3.40	36.1702	32.6924	29.5652	26.9841	24.8175	22.9726	21.3836	20.0000	18.7845	17.6165	16.5048
3.50	37.2340	33.6539	30.4348	27.7777	25.5474	23.6483	22.0125	20.5832	19.3970	18.1346	16.9803
3.60	38.2979	34.6154	31.3044	28.5714	26.2774	24.3243	22.6415	21.1764	19.8895	18.6527	17.4757
3.70	39.3617	35.5770	32.1740	29.3650	27.0073	24.9999	23.2704	21.7647	20.4420	19.1709	17.9611
3.80	40.4255	36.5386	33.0436	30.1587	27.7372	25.6755	23.8993	22.3529	20.9944	19.6891	18.4465
3.90	41.4894	37.5000	33.9132	30.9523	28.4671	26.3511	24.5232	22.9412	21.5469	20.2078	18.9320
4.00	42.5532	38.4616	34.7828	31.7460	29.1971	27.0268	25.1572	23.5294	22.0994	20.7254	19.4175

	21	22	23	24	25	26	27	28	29	30
2.00	9.1743	8.7236	8.2645	7.8431	7.4627	7.1174	6.7797	6.4725	6.1728	5.8997
2.10	9.6330	9.1703	8.6777	8.2353	7.8358	7.4733	7.1186	6.7961	6.4814	6.1947
2.20	10.0917	9.6070	9.0909	8.6275	8.2090	7.8292	7.4576	7.1197	6.7901	6.4897
2.30	10.5505	10.0437	9.5041	9.0196	8.5821	8.1851	7.7966	7.4434	7.0988	6.7847
2.40	11.0092	10.4804	9.9174	9.4118	8.9552	8.5410	8.1356	7.7670	7.4074	7.0797
2.50	11.4679	10.9171	10.3306	9.8039	9.3284	8.8969	8.4746	8.0906	7.7161	7.3447
2.60	11.9266	11.3537	10.7438	10.1961	9.7016	9.2527	8.8136	8.4142	8.0247	7.6697
2.70	12.3854	11.7904	11.1570	10.5882	10.0747	9.6085	9.1526	8.7378	8.3334	7.9647
2.80	12.8441	12.2271	11.5702	10.9804	10.4479	9.9644	9.4915	9.0615	8.6420	8.2596
2.90	13.3028	12.6633	11.9834	11.3725	10.8209	10.3202	9.8305	9.3851	8.9507	8.5546
3.00	13.7615	13.1094	12.3967	11.7647	11.1941	10.6761	10.1695	9.7087	9.2593	8.8496
3.10	14.2202	13.5371	12.8009	12.1569	11.5672	11.0320	10.5085	10.0323	9.5680	9.1446
3.20	14.6789	13.9738	13.2231	12.5490	11.9404	11.3879	10.8475	10.3559	9.8766	9.4396
3.30	15.1376	14.4105	13.6363	12.9412	12.3135	11.7437	11.1864	10.6796	10.1852	9.7345
3.40	15.5964	14.8472	14.0466	13.3393	12.6966	12.0996	11.5254	11.0033	10.4938	10.0295
3.50	16.0550	15.2843	14.4628	13.7264	13.0598	12.4555	11.8644	11.3269	10.8024	10.3245
3.60	16.5137	15.7205	14.8760	14.1176	13.4329	12.8114	12.2034	11.6505	11.1111	10.6195
3.70	16.9724	16.1572	15.2392	14.5098	13.8060	13.1673	12.5424	11.9741	11.4197	10.9144
3.80	17.4311	16.5939	15.7025	14.9020	14.1792	13.5231	12.8814	12.2977	11.7238	11.2094
3.90	17.8899	17.0306	16.1157	15.2941	14.5522	13.8790	13.2204	12.6213	12.0370	11.5044
4.00	18.3486	17.4673	16.5289	15.6862	14.9264	14.2349	13.5593	12.9450	12.3457	11.7994

Example.—For a juice extracted of 116° Baumé and a lime addition of 3%, the quantity of milk of lime of 20° Baumé to be added per hectolitre of juice is 1443.1 = 12.458 litres of milk of lime.

*Details of the double carbonation process.*—The double carbonation process as conceived by Possoz the chemist, Perrier the engineer, and Cail the constructor, is carried out as follows:—

1. *Liming.*—Milk of lime is added to the *cold* juice immediately it comes from the diffusion battery. The juice should not remain exposed to the air without being limed, as it is apt to ferment.

The liming is generally done in a carbonation pan, but, what is better still, sometimes in a special vessel provided with an agitator. From 1.5 to 3 per cent. of lime reduced to milk of lime is added. (See Unger's table on p. 77.)

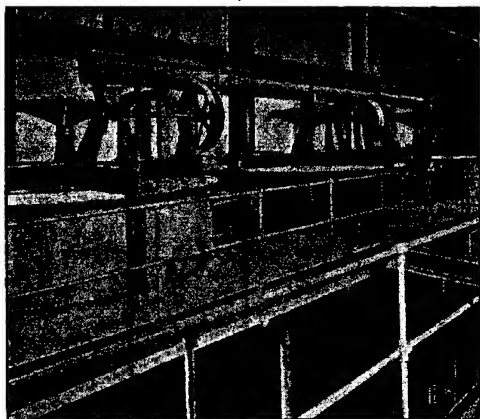


FIG. 29.—Special vessels for treating the beet juice with milk of lime (CAIL).

2. *Injection of carbonic acid.*—*Froth prevention.*—As soon as the milk of lime is added to the juice carbonic acid is injected. A thick froth forms, which is kept down either with a fatty substance, carbolic acid, or a solution of camphoriferous oil in alcohol, which causes the balls of juice, filled with gas, to glide rapidly the one over the other and to split them rapidly, or with froth preventers (p. 89).

3. *Application of heat.*—When carbonation has had a good start heat is applied by a steam coil which makes several convolutions round the pan, and the steam so regulated that the juice reaches a temperature of 75° to 80° C. (167° to 176° F.) by the time carbonation is nearly completed.

*The final point of the process, and signs by which it is recognised.*—

The near completion of the process is indicated by the disappearance of the froth and by the special colour which the liquid assumes. The workman then diminishes the injection of gas, so as to be able to stop short at exactly the right point. Formerly this final point was easily ascertained. A little juice was taken in a big spoon, and examined in the light. When the carbonate of lime slowly separated from the juice, and precipitated on the bottom of the spoon, meandering over its surface, leaving a supernatant bright straw-yellow juice, the operation was finished. If the precipitate took too long in forming, the operation was not yet finished. But, if the carbonate precipitated too quickly, and the juice was of a brown colour, the saturation point had been exceeded.

*Chemical control.*—The spoon test is now controlled by chemical analysis. According to the nature of the beet, the amount of lime to be left in the juice at the end of the operation is fixed beforehand, say 0.1 to 0.2 per cent. The process is stopped when this point is exactly reached. The workman has a small laboratory on a table in front of his pans, which enables him very rapidly to test the amount of lime in the juice (see p. 98). Possoz elaborated a very simple system of analysis, but which analytical progress superseded. But it is to him that the chemical exactitude of this first carbonatation is due. When the exact point is reached the gas arrival tap is closed and the steam increased, so that the juice attains a temperature of about 95° C. (203° F.). Then the steam is turned off, and the pan immediately emptied into a vessel beneath. The juice formerly remained in the vessel, into which it was decanted, until the muddy precipitates were collected at the bottom. The clear supernatant juice was drawn off through an elastic pipe, the head of which floated on the liquid, supported by a buoy. When the juice commenced to come away turbid the decantation process was stopped, and the muddy residue was sent to the filter presses.

*Filtration of liquor from first carbonatation.*—Nowadays the turbid juice, from the carbonatation process, is immediately sent to the filter presses without deposition followed by decantation. The juices to be subjected to the second carbonatation are thus more clear and not nearly so turbid, a great advantage, as the mud of the first carbonatation contains the major portion of the impurities of the juice, and all the mud which escapes from the first carbonatation, and passes into the second, is so much impurity redissolved in the juice by the complete saturation by  $\text{CO}_2$  about to be imparted to it.

*The second stage.*—The clear juice from the first carbonatation is thus sent on to the second carbonatation process. Lime, as milk of lime, is added to the extent of two to ten-thousandths according to the nature of the beets.  $\text{CO}_2$  gas is then injected to completely saturate the lime, the juice is brought to 100° C. (212° F.), and the

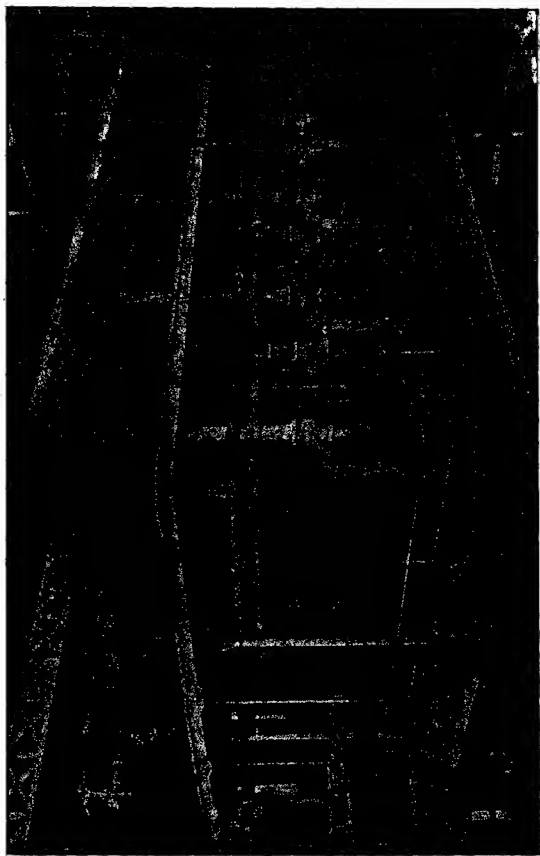


Fig. 80.—Intermittent carbonation tanks (Carr).

operation finished. This turbid juice is immediately run into settling tanks, or passed to the filter presses, like the first carbonatation juice. The limpid juice is now sufficiently pure to be evaporated and boiled down to strike point. The cold system of carbonatation described above was especially necessary in the case of *pressed juice*.

*Modifications in carbonatation process due to more hot and purer diffusion juice.*—Since diffusion became general certain alterations have been made in the carbonatation process. Diffusion juice has already been raised to a rather high temperature in the diffusers, viz., 75° to 80° C. (167° to 176° F.), and again, owing to the method of its extraction, it is purer than the pressed juice. Osmosis has already kept back certain substances which would naturally enter with the juice into the carbonatation process, leaving behind in the beet cell a large proportion of the albuminoid and pectic bodies, which the pulping machines and presses liberated, so that they passed into the juice. A large proportion of the impurities present in pressed juice are thus absent from diffusion juice.

*The purity of the juice the cause of the abandonment of diffusion in the first instance.*—It was really because diffusion juice is purer than pressed juice that Mathieu de Dombasle had to abandon his maceration process. The juice he obtained by maceration could not be suitably defecated for want of albumen. Even when certain beet juices were treated by Rousseau's process, defecation was so difficult that it was often necessary to add blood, or white of egg, or even cow's milk, to form a coagulum to envelop and carry down impurities.

*Many precautions indispensable with pressed juice superfluous in the case of diffusion juice.*—Some of the precautions necessary with pressed juice, e.g., conducting the first carbonatation in the cold, are quite unnecessary with diffusion juices. Moreover, there are some advantages in working in the hot condition, as the pectic and albuminoid bodies, existing in smaller proportion, then coagulate better in presence of lime.

*Liming, temperature, and proportions.*—*German versus French practice.*—The diffusion juice is limed in a mixing vessel (Fig. 29), then heated to 80° C. (176° F.) in a special reheater, and enters into the first carbonatation in the hot state. In certain countries, more especially in Germany, it being thought unnecessary to use so much lime with diffusion juice as with pressed juice, they diminished the quantity. But instead of limiting themselves to a proportionate reduction, they went to the opposite extreme, and did not add enough lime. The result of this was that in Germany, instead of two saturations it has been necessary to make three, because the first and only important one was defective. In France, however, they add plenty of lime in the beginning, which is no doubt the right method of working. The second carbonatation then suffices.

*Other methods.*—Efforts have been made to find other and possibly more preferable processes. Other defecating agents have thus been discovered, which are (1) used as aids to carbonatation, and (2) sometimes conjointly thereto.

*Carbonatation by means of baryta.*—Baryta is the defecating agent *par excellence*; it is the first and most active element of the series of alkaline earths—baryta, strontia and lime. Strontia comes after it, and lime the last of the three. Lime, however, is preferred, on account of its abundance in nature, its easy conversion from carbonate into quicklime, and last, but not least, its cheapness, and the ease with which it is handled.

*Baryta far too costly, and its preparation insanitary.*—Baryta, however, is sufficiently abundant as sulphate (*barytes*), but the sulphate is only transformed into caustic baryta with great difficulty, and disengages so much sulphuretted hydrogen in the process that its manufacture is banished from all inhabited places. Manufacturers are thus thrown back on the less abundant carbonate of baryta, witherite, from which to make their caustic baryta.

*Baryta too costly by itself.*—Attempts have been made to use baryta by itself, but its cost is too great, and the revivification of the carbonate contained in the sludge is not profitable enough.

*A sufficiently cheap and effective auxiliary in the second stage.*—Baryta is now sometimes used as an auxiliary to lime in the second carbonatation, to precipitate all the impurities which the lime was unable to remove in the first process. For this purpose baryta is very useful without running too dear.

*Carbonatation in the diffusers by soda crystals and barium hydrate.*—Another process consists in adding to the diffusion 1 lb. of carbonate of soda per 1,000 lb. of beets, thus perfectly neutralising the juice; then, as soon as the juice is drawn from the last diffuser cell, 1 lb. of crystallised baryta stirred up in water is run into the vessel. This forms an abundant precipitate, and the lime is finally added without troubling about the baryta. The carbonatation is effected as usual. This method, they say, likewise gives excellent results.

*Fears as to the toxic dangers incidental to the use of baryta.*—The danger attendant upon the use of baryta on account of its poisonous nature has been debated. This fear is, however, regarded by some as groundless, because baryta precipitates are so insoluble that not a trace of this alkaline earth remains in the saccharine juices. But it may be urged that objection was taken to the use of basic acetate of lead under similar circumstances. During the season 1899-1900 seventeen French beet-sugar factories used baryta as a defecating agent.

*Strontia is not used as a defecating agent.*—Strontia is not used in defecation, but finds a considerable use in the treatment of molasses, and forms the basis of a new method of extracting sugar therefrom.

TABLE XXIII.—SOLUBILITY OF STRONTIA, STRONTIUM HYDRATE, AND CAUSTIC BARYTA IN WATER.

Temperature °C.	Per Cent. SrO.	Per Cent. Sr(HO) <sub>2</sub> + 8H <sub>2</sub> O.	Per Cent. BaO.	Temperature °C.	Per Cent. SrO.	Per Cent. Sr(HO) <sub>2</sub> + 8H <sub>2</sub> O.	Per Cent. BaO.
0	0.95	0.90	1.50	55	2.54	6.52	14.71
5	0.41	1.05	1.75	60	3.08	7.77	18.76
10	0.48	1.23	2.22	65	3.62	9.29	24.67
15	0.57	1.46	2.89	70	4.35	11.16	31.90
20	0.68	1.74	3.48	75	5.30	13.60	56.85
25	0.82	2.10	4.19	80	6.56	16.83	90.77
30	1.00	2.57	5.00	85	9.00	23.09	...
35	1.22	3.13	6.17	90	12.00	30.78	...
40	1.48	3.80	7.36	95	15.15	38.86	...
45	1.78	4.57	9.12	100	18.60	47.71	...
50	2.13	5.46	11.75	101.2	19.40	49.75	...

*The machinery and plant used in treating the juice.*—The measuring tank.—When the juice leaves the cells it runs into a cistern called the measuring or gauging cistern, in which the amount of juice drawn from the diffuser is measured, and tested to secure uniformity in the density of the liquid and control the proper working of the battery itself. Measuring cisterns vary in number and size in different countries. In France there is generally only one. Certain factories, however, have two, one which is being *emptied* whilst the other is being *filled*. This is a mistake, because it in no way forwards the work when the measuring tanks are well designed and arranged. All that is required is that the cistern should empty itself rapidly through a wide pipe. This renders the employment of a second one superfluous.

*The special measuring tanks in use in Belgium.*—In Belgium the duty is collected on the juice drawn from the battery, and the quantity is measured in this cistern. It accordingly is of a special shape. It is a tank resting on feet with levelling screws, in such a way that it is quite horizontal. It is filled to overflowing all round, so as to make sure of the juice drawn off. The liquid runs over into a gutter, which discharges into a close vessel. The cistern itself is closed by a lid, and by resistant metallic wire gauze. The closed vessel, the lid, the taps are all under lock and key, and no one can touch them without the concurrence of the exciseman, who notes each time the density, which is taken by means of a special automatic test-glass, likewise under key. There is thus no possible



means of fraud, and the manufacturer is exactly informed as to the amount of juice drawn off. In France the information upon this point is not precise, for in spite of the float and indicator, if the workman does not examine these at the right moment there is no

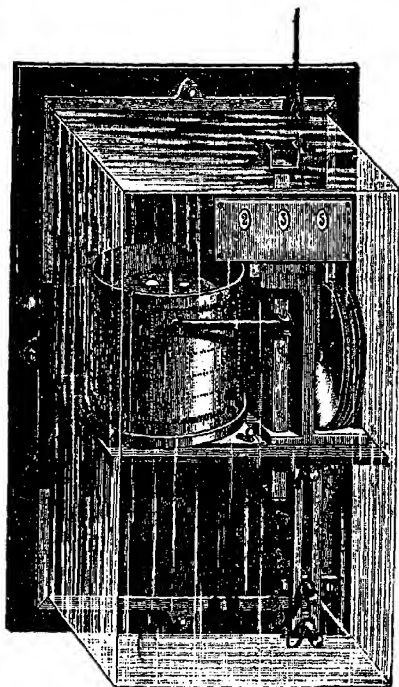


FIG. 31.—Horsin-Deon's automatic register.

guarantee as to the regularity of the work. To remedy this, Horsin-Deon designed his automatic register.

*Horsin-Deon's Automatic Register.*—A cylinder rotated horizontally by clock-work carries a roll of paper divided into hours, quarter-hours, and fractions of five minutes, a pencil car attached

to an arm bears directly upon the paper; this pencil, moved by a float, follows, and registers every change of level in the measuring tank. It will thus be seen that this instrument records every change that takes place in the measuring tank, whether it be charging, discharging, or irregularities of the work of any kind. The lines traced from the lower to the upper part of the paper show the charging of the measuring tank; and *vice versa*, those traced from above downward the discharging; a horizontal line indicates a stationary level. A counter records the total number of cells whose juice has been drawn off. M. Horsin-Deon has so arranged this apparatus that it may be located at a distance from the diffusion battery, preferably in the office or laboratory. In order that the work may be regular and the extractions uniform, two electric bells are connected with the apparatus, and indicate the proper moment to open or close the battery valves. With this apparatus, where a complete record of the diffusion work is automatically made, one can obtain absolute certainty that all orders from the laboratory or office have been strictly carried out, and that time has not been lost at certain hours of the night only to be made up by hurried work in the morning. The record page is changed every twelve hours.

*Automatic sampling of the juice from the diffusers.*—The easiest arrangement for automatically sampling the juice from the diffusers consists of a three-way valve, opened and shut by the rise or fall of a float in the measuring tank. One of the openings of the valves communicates with a stand pipe extending above the greatest height to which the tank is ever filled, whilst the second opening connects the stand pipe with the bottle, in which the sample is to be collected. The float is so adjusted with the stem of the valve that when the juice reaches a certain height it lifts and opens the valve, and places the stand pipe in communication with the tank. When the level in the tank falls, the opening from the stand pipe to the tank is closed, whilst at the same time that connecting the stand pipe and bottle is opened, and the juice which filled the tube passes into the bottle. This is repeated every time a cell of juice is drawn, and furnishes a sure and accurate sample. A certain amount of basic acetate of lead, proportional to that used in analytical work, is placed in the bottle to preserve the juice. Before analysis sufficient acetic acid is added to the sample, to give a decided acid reaction. An aliquot part of the sample is taken for polarisation and the determination of the glucose. Before making the glucose determination the lead should be precipitated and removed by filtration. The opening in the three-way valve should be at least half an inch in diameter, to prevent clogging. The tube leading from the tank should also be provided with a fine strainer. So that the valve may work quickly enough to prevent placing the bottle in communica-

tion with both tube and tank at the same time, it should be fitted as follows: A section of the valve through the openings should exhibit a T-shaped groove, so that a quarter-turn may suffice to connect the stand pipe with either the sample bottle or the measuring tank. The stem of the valve should be prolonged and fitted with

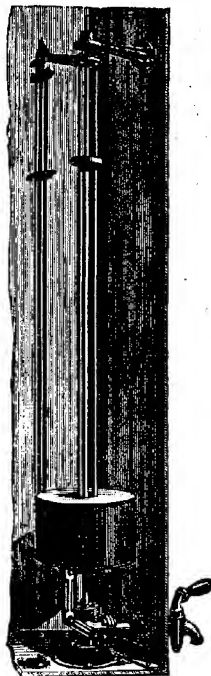


FIG. 32.—Apparatus for sampling automatically the juice from the diffusers.

a pinion  $\frac{5}{8}$  inch in diameter, which in turn engages a spur-wheel 4 inches in diameter. The spur-wheel shaft is fitted with a drum 8 inches in diameter. The wire extending from the float makes a couple of turns round this drum, and is then weighted. The float is so arranged that it has a rise or fall of about 1 inch. The entire apparatus, provided with a suitable frame, is kept under lock and

key. The delivery tube from the stand pipe extends nearly to the bottom of the sample bottle, in order that the stream of juice may thoroughly mix with the basic acetate of lead and with preceding charges. The dimensions of the gearing and drum given are such that a very slight change in the level of the juice in the measuring tank will open or close the valve.

*Eugene Langen's automatic register.*—This apparatus, designed by Eugene Langen of Cologne, is intended not only to register the measurement of the juice, but also to determine its density. The measurer of the volume of the juice drawn consists essentially of a copper cylinder containing six compartments, and is similar in its

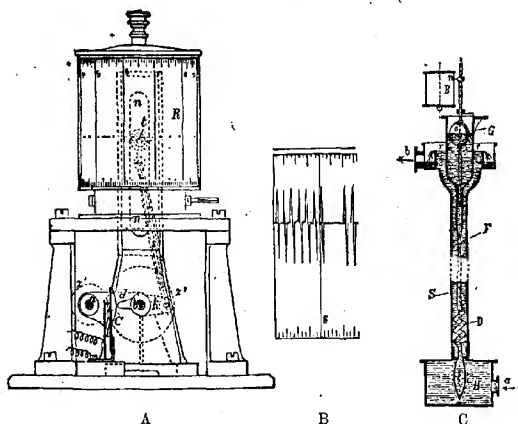


FIG. 33.—Eugene Langen's automatic register of the volume of juice drawn from the cells.

action to a gas-meter. The juice from the cell passes immediately into the measurer, thence to the carbonatation or clarification. The quantity of juice is indicated in cubic metres by means of the counter on a cylinder R. To accomplish this the counter communicates with the axis, which makes a complete turn for each diffuser of juice drawn. The axis *a* transmits its motion to the toothed wheels *s*<sup>1</sup> *s*<sup>2</sup> (variable at will) to the axis *b*, upon which is fixed a crank *k* and a projecting arm *d*. The crank, by means of the connecting rod *s*, raises or lowers the waggon *t*, carrying a pencil and travelling on the guides *n n* in such a manner as to trace a diagram on the slowly revolving cylinder R, which is driven by clock-work.

The arm *d*, touching the electric contact *e*, closes the circuit and rings an electric bell, which notifies the batteryman that he must close the juice valve. Fig. 33 B indicates the form of the diagram traced. The lines in the diagram which are very nearly vertical indicate the time required to discharge a diffuser of juice. The short horizontal lines show the length of times between discharges of juice and their height, above the centre line shows whether the correct amount of juice was drawn. The automatic determination of the density of the juice depends upon the principle of communicating vessels. A column of juice of an invariable height counterbalances a column of water whose height is proportional to the density of the juice. A portion of the juice measured by the meter passes through the small reservoir H, into a tube S, provided with an overflow at *r*. Inside the tube S is another tube F D, which terminates above in a funnel-shaped vessel and below in a flexible rubber bulb F. The interior of this tube, including the bulb, is filled with water, whose height is registered on cylinder *b* by means of a float carrying a pencil. The variable temperatures of the juice have no influence on the apparatus, provided the column of water is of the same temperature as the juice surrounding. For this reason the tube F is spiral at the lower end. The specific gravity of the juice so obtained is reduced to the normal temperature, and the degrees Brix or Baumé noted. Langen has substituted a bundle of very fine copper tubes for the spiral, in order to more readily equalise the temperature of the juice and water. Foam and mechanical impurities do not affect the apparatus. If used independent of the automatic measurer, a double ball valve must be used to prevent wastage from the overflowing of the juice.

*Carbonatation Tanks.—Liming in the measuring tanks.*—The juice is sometimes limed in the measuring tanks, but chemical control then becomes impracticable. It is better to pump the juice to special tanks (Fig. 29).

*The carbonatation tanks, their original form and defects.*—The carbonatation tanks vary in size and shape. In France the first tanks were square, wide and shallow, and the juice occupied a height of 20 to 25 inches. The gas was injected into the juice through a perforated coil in the bottom of the tank.

*Shallow versus deep carbonatation vessels.*—This was a bad idea. To utilise it to its fullest extent the carbonic acid gas should bubble through a great depth of liquid.

*The deep form of carbonatation vessels adopted in Austria.*—In Austria they now use cylindrical vessels containing a great depth of juice. French manufacturers now recognise this is the better method of working. *Square deep tanks* (Fig. 30) are therefore now used, the *square form* being preferred because the series of tanks then occupy less space. The depth of the juice is about 40 inches.

*Prevention of frothing during carbonation.*—To get rid of the froth formed at the beginning of the process, so voluminous that it runs over the sides of the tank, if a fatty body, etc. (p. 78), be not added, *froth eliminators* have been devised. Froth eliminators are of two kinds. The first, known as Evrard's *emousseurs*, consist of a sheet of steam projected on the surface of the liquid, which breaks the froth. This is done by a pipe perforated with small holes placed horizontally above the liquid. Steam is run in at high pressure, which escapes with force through the whole of the small apertures simultaneously. Froth preventers are also used, based on the hydraulic turnstile principle, through which steam is passed. These two appliances act well but consume a great deal of steam, and have been generally replaced by mechanical froth preventers—shafts driven mechanically, and armed with blades which beat up the froth and destroy it.

*Froth curbs like those of a soap pan.*—A continuation or enlargement high enough to prevent the froth from overflowing acts better than froth preventers. These enlargements, from 6 to 10 feet in height, are closed by a lid surmounted by a very wide pipe passing through the roof (Fig. 35). All annoyance and expense of froth preventers are thus got rid of. Robert of Seelowitz first employed rational pans of great depth and enlargements of considerable height in Austria. Since then he has been copied everywhere.

*The carbonic acid injection piping.*—Inside each carbonation tank, resting upon the bottom of the tank, is a system of perforated piping, either in the form of a square, a circle or a star, for the injection of carbonic acid gas. The tanks are heated internally by a steam coil, circulating right round the interior of the tank, or by the radiating heater (Fig. 36). They are emptied through an aperture inserted in the lowest part of the inclined bottom, which is closed by a plug during working hours. The tank is discharged either into a settling tank or into a mixing tank with an agitator in front of the pump, which pumps the carbonated juice to the filter presses. The second carbonation tanks do not essentially differ from the first; but they are not fitted with froth preventers, or if so they are very simple, because the juice, heated a second time, froths much less than previously.

*Continuous carbonation.*—The carbonation tanks work intermittently, and would cause much loss of time if a numerous series of these pans were not kept at work, *two at least being idle—one being charged, and the other being emptied.* Protracted attempts were made to produce a system of continuous carbonation. Overflow tanks were therefore constructed, and the gas injected in an opposite direction to that of the juice. But there is no absolute fixity in the percentage of the lime left in the juice treated. Again, the juice has been passed through pipes in an opposite direction to the gas,

and the same result obtained, so that, after all the attempts, not one has survived in practice. But Horsin-Deon caused both the gas and the juice to pass in the same direction, so that both the carbonic acid in the gas and the lime in the juice diminish simultaneously. When the appliance is adjusted and regulated, the small differences in the amount of lime used and in the richness in  $\text{CO}_2$  of the gas, produce but little alteration in the amount of lime in the juice. Horsin-Deon's appliance acts like Liebig's potash bulbs, in the form of a filter press, with a series of narrow chambers succeeding very wide ones, so in the latter the bubbling of the gas is highly divided. The limed juice and the gas flow in at one end and out exhausted at the other. When the apparatus gets soiled with mud, it is cleaned by dismantling it like a

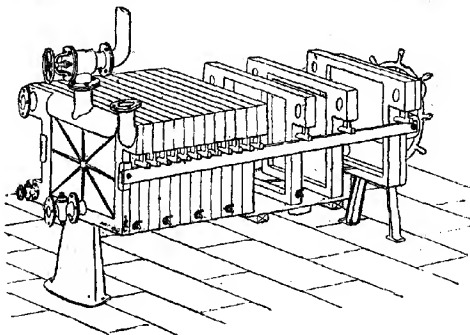


FIG. 84.—Horsin-Deon's continuous carbonator.

filter press. But these cleanings are not frequently required. It occupies little space compared with ordinary carbonatation pans.

*Gibert's continuous carbonatation process in rectangular pans.*—This system comprises two rectangular pans, 1 metre to 1.2 metre in width by 3.5 metres in height, say 4 feet wide by 12 feet in height. These two pans are placed parallel, in the direction of the length, have each an inclined bottom, sloping in a contrary direction, so that the juice running through them in the direction of their length passes from the one to the other. In this 7 metres (say 24 feet) journey the juice meets, in each pan, special gas jets which distribute the gas in the mass of the juice. As the level of the juice flowing away through a funnel is kept at a height of 2.5 metres, say 8½ feet, absorption is complete and carbonatation instantaneous.

*Precautions.*—Certain precautions have to be taken in the working of continuous carbonatation. These are as follows: (1) The

liming should be regular; liming by powdered or lump lime is advisable. (2) The pressure of the juice, as it flows into the tank, should be constant. The pan must not be fed by a pump, but preferably from a full cistern provided with a pressure regulator. If

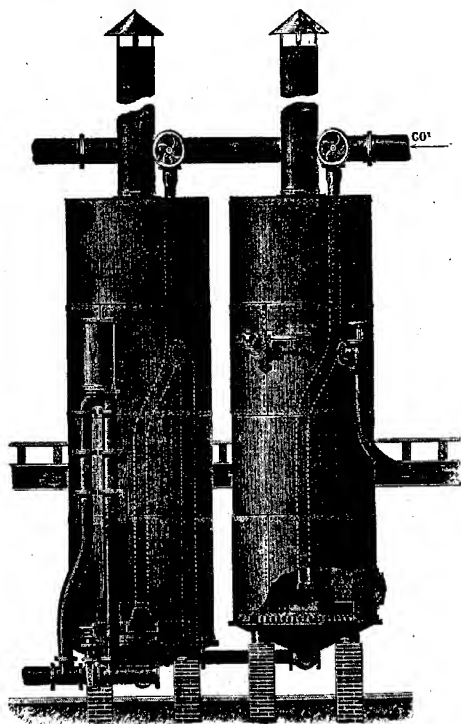


FIG. 35.—Gibert's continuous carbonatation process (as applied to cylindrical pans by Maguin).

these two conditions be fulfilled the carbonatation is effected regularly, and the carbonatation workman has nothing more to do than to regulate the gas-tap according to the results he gets on testing. The advantages claimed for Gibert's system are—space, economy, absolutely regular working, considerable output, perfect



utilisation of the carbonic acid. Maguin has applied Gibert's system to cylindrical pans, as shown in Fig. 35. The juice is fed into the second pan either from the top or by means of a vertical pipe, starting at the bottom from the communication pipe, and ending at the top in an elbow. In the second pan, at a level slightly higher than the level of the juice, or through the bottom even of this second pan, taps placed in the right position enable the bottoms of the pans to be flushed so as to avoid any obstruction. A Desmet's froth preventer may be adapted to the first pan.

*The radiating heater applied to carbonatation.*—Not only does this apparatus (see p. 183) allow the carbonatation juice to be heated in the pan itself by the juice steam or the escape steam, but it also perfectly divides the juice and the gas, and produces a complete emulsion from their interaction. The upper sloped partitions of the radiating heater are therefore prolonged by two wrought-iron baffles, which extend almost to the sides of the pan so as to force the gas to pass, in a very divided form, in an inverse direction, and thus form a very energetic circulation. Carbonatation is effected rapidly and well.

*Manufacture of carbonic acid and quicklime.*—In Rousseau's process carbonic acid was produced by burning charcoal. A pump drew the gas forward and pressed it into the limed juice. Sugar manufacturers had to buy lime or have a lime kiln in their factory.

*The charcoal furnace superseded by the lime kiln.*—But one day, very naively says Horsin-Deon, they were struck with the idea that burning lime in kilns produced carbonic acid, and then and there set to work to extract the carbonic acid evolved from the top of the lime kiln, Rousseau's charcoal furnace being set to one side. Sugar factory lime kilns differ somewhat from ordinary ones. They are generally much larger in proportion to the size of the factory. As a rule, there is only one kiln, or at the most two, to each factory, because it is difficult to regulate identically the working of several furnaces. It is on the continuous principle, that is, burning is not stopped for charging and re-charging: it is charged from the top and discharged from the bottom.

*The method of heating the furnaces of the lime kiln.*—Two methods of heating are adopted. The one consists in having hearths underneath, on which a coke fire is continuously kept burning; in the other the coke is fed into the kiln with the lime, and there are no heating furnaces or hearths in which fuel alone is burnt.

*The combination system of heating the kilns.*—Sometimes, however, the two systems are combined, and not only is the fuel mingled with the lime, but heating furnaces are also used.

*The several methods of heating equally preferable.*—If manufacturers are questioned, says Horsin-Deon, as to the best of the three systems, little satisfaction is got, each one praising the system which he adopts.

They therefore are equal. He prefers the second method as being the most simple.

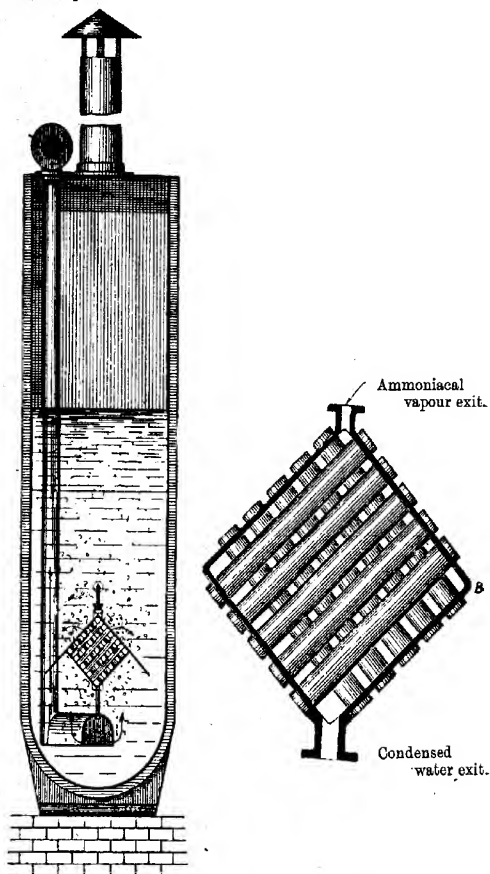


FIG. 86.—The radiating heater applied to a carbonatation pan.

*The fuel should be free from sulphur and ash.—The coke used should be as free from sulphur and ash as possible, the sulphur*

giving rise to sulphurous acid, which militates against the successful working of the carbonation process, whilst the ash introduces impurities into the lime which contaminate the juice.

*Necessity for selecting a pure limestone.*—The limestone ought to be as pure as possible, and so compact as to produce lumps of well-burnt lime which do not split up when taken out of the furnace. It should contain neither chlorides, sulphates, magnesia nor clay.

*Analysis of limestone.*—1. *Moisture.*—Dry 5 grammes over the blue flame of an argand Bunsen. Loss multiplied by 20 equals moisture per cent.

2. *Insoluble silicious and organic matter.*—Five grammes are boiled in 100 c.c. water and 20 c.c. hydrochloric acid for 10 minutes, evaporated to dryness to render silica insoluble, taken up with water; the insoluble is collected on a previously thoroughly dried filter, the weight of which has also been ascertained beforehand. The filter and its contents are washed and dried until of constant weight. This weight—minus that of the filter—multiplied by 20, equals amount per cent. of insoluble silicious matter plus organic matter.

3. *Insoluble silicious matter.*—The filter and its contents from 2 are ignited; the weight of the residue, multiplied by 20, equals per cent. insoluble silicious matter.

4. *Organic Matter.*—The difference between the nett weights obtained in 2 and 3, multiplied by 20, gives the organic matter per cent.

5. *Sulphuric acid.*—An aliquot part after solution is brought to the boil, barium chloride added, the sulphuric acid estimated in the usual way and brought to per cent.

6. *Oxides, etc., of iron and alumina.*—An aliquot part of filtrate from 2 is oxidised by nitric acid and ammonium hydrate added, the precipitate washed, dried and ignited in the usual way and brought to per cent.

7. *Lime* is estimated in the ordinary way in filtrate from 6 by precipitation with ammonia and ammonium oxalate and brought to per cent. of  $\text{CaCO}_3$ .

8. *Magnesia* is estimated in filtrate from 7 by precipitation with hydric disodic phosphate, the precipitate filtered, washed with ammonia water, ignited and weighed, calculated to magnesium carbonate, and brought to per cent.

9. *Iron* is estimated in 10 grammes by dissolving in hydrochloric acid and titrating with decinormal solution of bichromate of potash; the result is calculated to per cent. of ferrous carbonate.

10. *Chlorine* is estimated by dissolving 5 grammes in nitric acid and treating with argentic nitrate in the usual way.

11. *Carbonic acid* is estimated by difference or in Schrotter's apparatus.

The limestone should contain a maximum of carbonate of lime

and a minimum of moisture—10 per cent. at the most. After the limestone has been burnt it is necessary to ascertain the quality of the quicklime which it yields, and the proportion of inert ingredients which it contains. This is done simply and effectively. To a certain amount of sugar dissolved in water, a weighed quantity of lime is added and the whole stirred. The sugar dissolves the lime, and leaves an inert residue. A more exact method could not be applied, as it is a repetition of what goes on in the factory. The works laboratory should adopt the same methods of testing as the substances will be submitted to in the actual routine of factory operations. That is the best way of ascertaining the quality of the raw materials, and of becoming acquainted with what goes on during the routine of factory work.

*The management of the lime kilns and purification of the carbonic acid.*—The kiln is charged with the limestone broken into lumps the size of the two fists. The charging of the kilns and the speed of the gas pump should be as regular as possible.

*The amount of coke per ton of limestone burnt.*—About 2 cwt. of coke is used for a ton of limestone. But the amount varies with (1) the good or bad construction of the kiln, and (2) the skill of the lime burner. The top of the kiln is hermetically closed by a trap, or reversed cone, which forms a plug. Above is a hopper or funnel into which the coke and limestone are charged. When the charge is ready the trap is opened, the cone is lowered and immediately closed.

*The rate of combustion is regulated by the speed of the pump.*—Underneath the charging door a small tunnel is arranged in the masonry, opening to the outside, and from this aperture there branches off a pipe, in communication with a pump of sufficient force to aspirate all the gases formed. The kiln works slowly or quickly according to the speed of the pump, which must therefore be so controlled that the kiln works in a regular manner, *i.e.*, with complete combustion of the fuel, and consequently *without the formation of carbonic oxide*, and without too much air in excess being carried through.

*The percentage composition of the kiln gas.*—The maximum theoretical richness of the gas obtained in this way is 44 per cent. of carbonic acid. But in actual practice the percentage varies from 25 to 30, never greater.

*The manner in which the kiln is fed.*—The limestone and the coke are raised to the top of the kiln by ingeniously constructed elevators, or by means of a simple rope and pulley. Baskets or trucks are used for this purpose.

*Gas scrubbers and coolers.*—Between the kiln and the pump the gas passes through one or several washers, which remove any ash or dust carried along in the train of the gas, and at the same time cool

it, for it is very hot, and as the work done by the pump is greater the cooler the gas, it is very desirable to cool it well. These washers or gas scrubbers consist of a vertical cylinder, divided into horizontal compartments by sheets of perforated wrought iron, to the number of four or five, according to the dimensions of the washer. The gas enters below and makes its exit from above. A current of water runs in an opposite direction from top to bottom; from which it escapes by a syphon. The perforated wrought-iron plates divide both water and gas, and cause a continual bubbling; moreover, a certain level of water is always maintained in the lower compartment, underneath which the carbonic acid enters through a perforated pipe. Sometimes the upper compartments are charged with coke, so as to divide the gases and the water better.

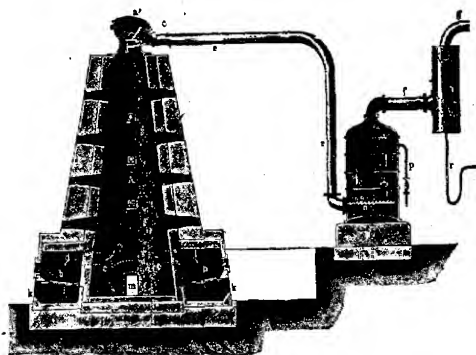


FIG. 37.—Beet-sugar works lime kiln and gas scrubber.

*Dust deposition chamber.*—A receptacle is placed, in the form of a rather larger chamber, between the kiln and the scrubber, in which the greater amount of the dust may be deposited. Another chamber is placed after the scrubber, in which any water accidentally carried forward by the gas may be deposited. This latter chamber also acts as an elastic obstacle to the strokes of the piston, which without it would make themselves felt both on scrubber and kiln.

*The gas-pump.*—The gas-pump is an enormous cylinder, in which a piston moves, but not at too great a speed. This pump has no valves, but a plain drawer or cylindrical distribution. Its discharge pressure is made directly into the carbonators. But as some, or even the whole, of the tanks may be idle, the end of the pipe carries a weighted valve, which rises and allows the gas to escape through

the roof when the consumption by the carbonatation pans is insufficient.

*Discharging the lime kilns, cooling and slaking the lime.*—The lime is taken from underneath the kiln, or from the side, according to the model of kiln. It is generally cold enough to be used at once. If discharged red hot, it has to be cooled. But this only occurs with badly constructed kilns or abnormal working. The lime is transferred from the furnace to the tanks, in which it is slaked by first moistening it with water, and then covering it with water until ebullition is over. When cold it is a thick paste.

*Converting the slaked lime into milk of lime.*—This paste is then run into mixers, which in a well-planned factory ought to be near the slaking tank. Sometimes, indeed, the slaking is done in the mixer itself. The lime mixers are cylindrical tanks (see Fig. 29), in which a vertical axis, provided with a rake of hanging chains, revolves. The chains, which rub along against the bottom of the tank, prevent any lime from accumulating there. The lime, in paste, is run into these tanks, along with water, and the rotation of the mixer produces a very intimate mixture called *milk of lime*.

*Running the milk of lime through a sieve.*—When the milk of lime is well made the contents of the mixers are run through a fine sieve, which separates the stones and the unburnt lumps, and a milk of lime, ready for immediate use, is now obtained.

*The proper density of the milk of lime.*—Matters are so arranged that the milk of lime marks 25° B., sp. gr. 1.205, the density most favourable to its successful employment. That corresponds to about 25 per cent. of lime; the exact strength is, however, controlled by analysis. By taking the milk of lime as containing 25 per cent. it will be necessary to use four times more milk of lime than the actual quicklime judged as being necessary for the carbonatation. If, for instance, it be desired to use 2 per cent. of lime per hectolitre of juice, 8 litres of milk of lime are used, or 8 British imperial gallons per 100 gallons (see Unger's table, p. 77).

*Gauging the milk of lime.*—The quantity of milk of lime added to the juice is gauged by a measuring tank, placed above the juice mixer, and the carbonatation tank, which the workman fills and empties by taps and special piping leading from the milk of lime reservoir, in which an agitator is constantly at work to keep the strength uniform.

*The necessity for care in connection with the whole liming process.*—All the work connected with the lime ought to be well managed and conducted, because on it depends the success of the carbonatation process. It is one of the most important adjuncts of the factory.

*The titration of the limed juice.*—Where the chemical control of the manufacture is systematic, the amount of lime in the juice is determined previous to carbonatation. But the lime is always esti-

mated at the end of the operation, both in the first and second carbonatation. As the determination has not only to be exact but rapid, the test is a volumetric one performed on the spot. In front of the carbonatation tanks is a small bench, holding all the apparatus for the test, which decides when the operation in any given tank is finished. The principal tool is a graduated burette or alkalimeter, fitted with a clip or stopcock, and containing the test acid. This test acid consists of sulphuric acid, diluted to such an extent that 1 c.c. = 0.001 of lime in the juice. A small quantity of the juice to be tested is filtered, and a known volume is measured out from it by means of a graduated glass on the vessel which is run into a test-glass. The test acid is run from the burette into the liquid, drop by drop, until the liquid is neutral. The number of c.c. used indicates the number of thousandths parts of lime contained in the juice. Litmus may be used as an indicator, but it is not quite sensitive enough for traces of lime; moreover, its colour is badly seen by night light. Litmus is blue in the presence of alkalies and red in the presence of acids and purple when neutral. Phenol-phthalein may also be used as an indicator; it is colourless in the presence of acids but purplish-red in the presence of alkalies. A few drops of a neutral alcoholic solution of phenol-phthalein are run into the liquor, which is immediately coloured red, and the acid then run in, with constant stirring; and when the liquor being tested becomes colourless the operation is finished. Rosolic acid is also used as an indicator, in which case the liquid is changed from red to pale yellow. It would appear to give better results than phenol-phthalein. But perhaps the best results of any are to be obtained from the use of methyl orange as an indicator. Alkalies turn it yellow, acids bright red. Only enough is used to tint the solution the palest of straw-yellows. When neutral it passes almost instantaneously to a rose. In titrations far too much indicator is generally used. Only the smallest quantity of the coal-tar colours named is required. In the case of phenol-phthalein it is well to bear in mind that ordinary methylated spirit is slightly acid. Sometimes the acid itself is coloured with the indicator, so that each drop gives its coloured reaction. The burettes are fixed by stands of different shape, and always include a rapid system of filling to the mark without making several attempts. Sometimes the test acid stock bottle serves as a support to the burette, so as to reduce the apparatus on the bench to a minimum. In fact, these simple tests are easily made by the carbonatation workmen, and tell them the point at which to stop. Carbonatation has thus become a regulated scientific operation, which is a great step in advance from the time when they used to trust to rule-of-thumb work.

To facilitate testing, whether in the sugar-works laboratory or in the works itself in front of the carbonatation pans, Sidersky has com-

bined the calcimetric apparatus into a twin burette. A is a revolving stand on foot carrying a rectangular frame in which are placed two Wolff's bottles B and *b*, with two mouths. This frame is surmounted by a stand, on which are fixed the two Mohr's burettes C and *c*, the upper parts of which end in bulbs. Each of the Wolff's bottles B and *b* has its mouth closed by indiarubber corks, each pierced with

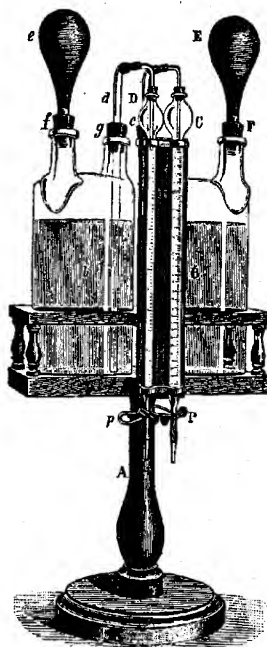


FIG. 38.—Sidersky's twin calcimetric burettes.

a hole. Through each of the corks F and *f* a tube attached to an indiarubber bulb E and *e* passes. The corks *g* are connected with the tubes D and *d*, which dip into the liquid and which communicate by means of small tubes bent at right angles and drawn out to a point with the burettes C and *c*. The burettes are closed by Mohr's clips P and *p*. The bottle B and the burette C contain the alkalimetric liquid,



the flask *b* and the burette *c* contain the soap test. The bottles *B* and *b* are filled by taking out the corks *F* and *f* connected with the bulbs *E* and *e*, which for the nonce are replaced by funnels. When the bottles are filled the corks are reinserted, care being taken to see that they are tight. By compressing the bulbs *E* and *e* the corresponding liquids ascend through *D* and *d*, and run into the corresponding burettes *C* and *c*. When the pressure is stopped *Dd* form a syphon and draw the excess of liquid from above the zero. The air of the burettes *Cc* escapes through small holes in the corks, closing the bulbs surmounting the burettes, thus avoiding any splashing of liquid. The burettes are graduated so as to show directly the percentage of lime expressed in grammes per hectolitre of juice. The alkalimetric test is made by Le Docte's solution<sup>1</sup> working on 25 c.c. The total lime is determined by means of Boutron and Boudet's normal soap-test solution working on 40 c.c. of juice in the usual way. The alkalinity of the carbonated juice, which is commonly determined as above, is generally expressed as quicklime ( $\text{CaO}$ ), although very often the alkalinity may be partially due to fixed alkalies or to ammonia resulting from the decomposition of organic matter by lime. No practical method has yet been discovered of estimating the free lime, but this is of no great concern to the manufacturer. What he wants to know is the *alkalinity* on the one hand and the *total lime* on the other, both expressed in the same way as  $\text{CaO}$ , so as to compare the two tests, the relation of which to one another is of great importance in the carrying out of the second carbonatation. The total lime may be estimated by Clark's soap test as modified by Boutron and Boudet. For proper chemical control each alkalimetric test ought to be followed by a "hardness" test, so as to see whether there are calcic salts in the juice or the syrup. This is not, however, done in actual practice, a few isolated "hardness" tests made in the laboratory being regarded as sufficient, whilst the alkalinity is being determined almost continuously. The carbonic acid used in carbonatation should contain a maximum amount of real carbonic acid, and neither carbonic oxide nor sulphurous acid.

The percentage of carbonic acid in the gas from the lime kilns is estimated as usual. The gas is collected in a tube graduated into 100 c.c. or any other equal divisions, then a piece of caustic potash is introduced into the water. The whole is shaken until absorption ceases. The volume of gas which has disappeared gives the percentage of carbonic acid. This figure may vary from 20 to 30. Special apparatus have been invented for executing this estimation. That of Possoz is the oldest; it consists of a large test-tube on foot

<sup>1</sup> Dilute 4.38 grammes  $\text{H}_2\text{SO}_4$  or 893 c.c. of normal acid to 1 litre. Working on 25 c.c. of juice, each c.c. = 0.1 gramme  $\text{CaO}$  per litre.

standing in a box in the shape of a cupboard. The absorption potash is in solution in a bottle, the contents of which are run forwards and backwards into a test-tube. The manipulation is therefore more simple than in the preceding method, and involves no risk to the operator's hands, which, in spite of all precautions, the potash attacks. Orsat's gas analysis apparatus is well known, and it should form part of the equipment of a sugar-works laboratory in the same way as it does of all other well-equipped laboratories.

*Determination of the purity of the carbonated juice.*—Although the "purity" in this case, as in all others, is only apparent, it should be determined with great care. The carbonated juice contains about 10 per cent. of dissolved substances, so that a difference of 0.1 in the dissolved matter may lead to an error of a whole degree in the purity, e.g., a carbonated juice with 10 per cent. of dissolved matter

and 8.5 per cent. of sugar will have a purity of  $\frac{850}{10} = 85$ . The same juice, with 10.1 per cent. of dissolved matter, will have a purity of  $\frac{850}{10.1} = 84.1$ ; again, if there only be 9.9 per cent. of dis-

solved, the purity will be  $\frac{850}{9.9} = 85.9$ . It is therefore necessary to determine the dissolved matter to the first decimal point at least by which the purity is determined only in whole degrees.

*Neumann's stone eliminator for sludge pumps or milk of lime.*—This machine, Fig. 39, is fixed on the aspiration pipe of the sludge or milk of lime pumps, so as to mechanically separate the juice from suspended matter, sand, stones and unburnt lumps, which have such a disastrous effect on the pump, pistons, cylinders and valves, without counting the obstruction to the channels of the filter-press frames. The juice enters, by an inclined tubulure, into the bottom of the machine, where it finds itself in rather a large space, in which heavy bodies, sand and stones, are deposited, then rising up the machine, it encounters a revolving sieve, with conical holes, through which it passes, freeing itself from any turbidity and suspended matters, after which it passes to a pump through the upper tubulure. The revolving sieve is constantly rubbed by a fixed scrubber, which removes the impurities adhering to the sieve. By a special arrangement it is possible to ascertain from the outside the height of the impurities inside the machine, and therefore when it is necessary to empty it, an operation which can be done very rapidly.

*Difficulty in early days of separating the lime sludge precipitate from the mother-liquor.*—The separation of the calcareous organic precipitate from the mother-liquor, after defecation and saturation, was for a long time difficult. The sludge or defecation deposits are

greasy, and do not filter through paper. Mechanical filters were badly adapted for the purpose. Again, carbonation, which produces a bulky deposit of sludge, could not be thought of without mechanical filters. Those interested revolved in a pernicious circle, which for a long time retarded all progress. Bag filters had been used in cane-sugar works, and efforts were made to utilise them for this purpose; but filtration in that way took far too long.

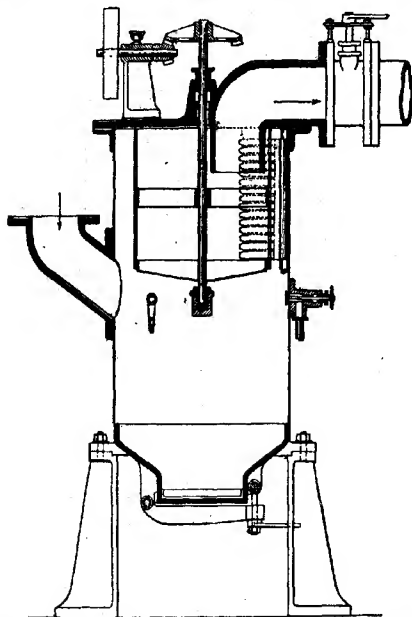


FIG. 39.—Neumann's stone-eliminator for sludge pumps and milk of lime.

*The history of the filter press.*—Howard thought of pressing the filters between frames, but the time had not yet come. It was not until about 1850 and 1856 that Teedham and James Kite constructed wooden apparatus capable of working somewhat efficiently. Finally, Danek replaced wood by cast iron. Trinks improved existing filters; and from 1864 filter presses became quite efficient under the hands of Hoekner, Roetger, Durieux, etc.

*Simultaneous introduction of carbonatation and filter press.*—The real filter press was thus born simultaneously with Rousseau's and the Perier-Possoz carbonatation process, the one reciprocally assisting the other in their simultaneous birth.

*The similarity in the filter presses of different present-day makers.*—Filter presses are now made by all engineering firms almost on the same principles and in the same sizes. There are two models, the larger of which, the monster filter press, is becoming more and more generally used.

*The general principle on which filter presses are constructed.*—The muddy liquid is forced under a pressure of two or three atmospheres into a square cloth bag pressed on its four sides by a double armature or plate which prevents it being deformed. As the bag is held on both faces by perforated plates at a determined distance, the liquid passes through the holes, and the calcareous mud remains in the interior, forming a cake as thick as the space between the perforated plates. From twenty-five to thirty, and even fifty, of these double frames are coupled together in the same appliance, thus forming the same number of bags into which the liquid is forced, and a filter press is thus obtained with a large filtering surface in a small space. All the bags are in communication with each other. Various makes only differ in the method of communication between the bags. Danek's filter presses were constructed as follows: The bags communicated with each other on the top. Each frame, therefore, had a wide hole in the centre of the top part. The cloths had holes in front of these apertures, then a frame carrying a sheet of perforated iron was fixed in the pile, then a cloth, then an empty frame, then a cloth, then at last a frame with its perforated iron plate. The hole of the empty frame communicated by smaller holes with the interior, and thus the muddy liquid entered and the cake became lodged in the same frame. The frames with perforated plates carried, moreover, one of these plates on each of its two faces so that it acted at the same time as the last frame of one bag and the first frame of the other. The fixed upright of the battery acted as the first frame, with perforated plate, and the last frame completing the battery also carried a plate; each of these frames was provided on both right and left sides with lugs, which rested on two horizontal pieces of iron, and the pressure of a strong screw kept everything in place. Trunks did away with the intermediate frame by making the frames which carry the perforated plate concave, and then the hole for the muddy juice was made in the centre of the plate and the cloths. The latter made a tight joint with the frames by means of large screws, through the centre of which the mud escapes. The other parts of the plant remain as before.

*Filter presses on the Dehne principle.*—The filter press invented by Dehne is the model most in vogue at the present time; and the

greater number of filter presses in actual use, not only in the sugar industry but also in every other industry in which filtration on the large scale is a prominent feature, are simply imitations of Dehne's type. His filter press is constructed on the same principle as Danek's, only all the frames have on the top and the side a lug, in which there is a passage through which the muddy liquid passes. A canvas washer, the same thickness as the filter cloths, serves as a joint between the opposite lugs. Nowadays these joints are perfected by indiarubber washers sunk into the cast iron. The plates of small

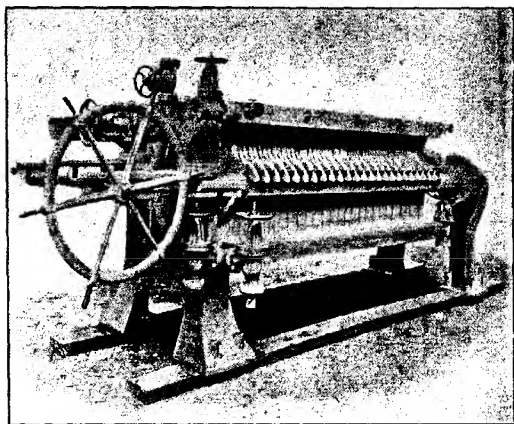


FIG. 40.—Beet-juice filter press (CAIL).

filter presses in the beet-sugar industry are 24 to 28 inches square with from twenty-five to thirty in a press, and hold about 10 cwt. of sludge. The plates of the larger-sized presses are 40 inches square, and form about eighty compartments. Double filter presses have been built, more especially in Austria, with 160 to 200 compartments. But these are merely two filter presses joined together end to end. The tightening up of these monster filter presses (Fig. 40), instead of being simply done by a screw, which would not be powerful enough, is effected either by hydraulic pressure or by a system of double lever arms acting through the agency of a movable screw.

*The montejus in connection with the filter press.*—The pressure on

the sludge in the filter presses is effected by a *montejus* or by a pump. The *montejus* is a stout cylindrical vessel into which the mud is run. A pipe in communication with the filter press leads down to the bottom of the vessel. When the cylinder is full, steam is applied through a tap in the top part of the vessel. The pressure of the steam causes the mud to rise up through the pipe and thus to fill the filter press. The whole contents of the vessel can thus be expelled and replaced by steam. When that has been done, the steam valve is closed, and a small side tap blow-off cock opened, through which all the steam contained in the cylinder is in turn expelled. The *montejus* is refilled with mud, and the process repeated. By using two *montejus* the work of the filter presses can be carried on uninterruptedly. The great drawback to the use of *montejus* is the enormous amount of steam consumed in keeping them at work. They have therefore been replaced by pumps. But the difficulty with pumps is in maintaining a regular pressure in the filter presses, which must not go beyond a certain limit.

*Accommodating or regulating the pump pressure to that required for satisfactory working of the filter press.*—Two methods are adopted to accomplish the end in view: (1) The first method consists in constructing the pump in such a way that the ratio between the steam cylinder and the pump cylinder should be such that the machine becomes inactive when the pressure behind the pistons of the pump reaches the desired degree. (2) The second method consists in using a sensitive regulator, which shuts off the steam when the pressure in the filter press is as great as desired. In well-constructed machines a combination of both methods is adopted. Moreover, the pump is double. There are thus two steam cylinders, and the same shaft serves for two couples, which are geared to this shaft in such a way that when one of the pumps is at the point of exhaustion the other is in full force. In this way the pump slows down, but starts again of its own accord as soon as the pressure lowers in the filter press.

*Washing the sludge.*—This is one of the most important points in the working of the filter presses. In fact, the cakes are wet with juice still containing much sugar, which it is, for economical reasons, necessary to remove as far as practicable. There are two methods of washing the cakes: (1) Washing the cakes in the filter press.

(2) Maceration of the cakes with water, and subsequent filtration.

1. *The first method fails by forming channels.*—The first method is that most often used. Generally, water under pressure is run into the space, through which the juice flows out during the first part of the process. All the frames, therefore, have taps underneath. In the beginning all those corresponding with the sludge frames are closed, and the others are opened. During washing, the opposite course is adopted. To prevent mistakes, the keys of these taps are

at different heights. But this method only partially eliminates the soluble matter. It consists, in fact, of only one water course, which simply conveys the water into the cakes, and then out by the tap in the adjoining plate. It will therefore be readily seen that the cake can only be but imperfectly washed, and, moreover, there is undoubtedly a great tendency for the water to form channels in the cakes and not to penetrate into and wash the other portions.

*Dehne's patent thorough extraction system.*—After the formation of the cakes the taps are all closed. The water is now forced, under a suitable pressure—preferably a somewhat higher pressure than the one at which the press has been filled—into a channel, and is distributed over the filtering surface of the alternate plates, one having a channel to the inside, the next none. It is now forced simultaneously over the whole surface of the cake, to penetrate the cake, the next cloth, to collect behind the filter cloth of the next plate, and finds its exit on the top of that plate, into the united efflux wash channel of every alternate plate. The air is allowed to escape through the channel, the valve of which is closed as soon as the matter comes out. It is claimed that the arrangement of these channels compels the fresh water to penetrate every part of the cake horizontally, and to take with it every particle of soluble matter. Generally the quantity of water to be forced into the filter press is decided upon beforehand. Sometimes washing is continued until a sample of the wash-water only shows  $1^{\circ}$  on the hydrometer (sp. gr., 1.0069). It depends on the routine and requirements of the factory. The wash-water goes to the second carbonatation. Sometimes the last portions are used to slake the lime.

*Extreme washing unprofitable.*—The sludge remaining in the filter presses after the filtration of the carbonated juice should only contain mere traces of sugar. A known weight is taken, washed repeatedly with boiling water, the wash-water filtered, carbonated, again filtered, the proper quantity measured and polarised. One should not expect to extract all the sugar from the sludge, as that would involve extreme washing, resulting in enormous volumes of very weak saccharine liquors, which it would never pay to concentrate, the working expenses being far greater than the values of the sugar recovered.

*Mashing or maceration of the sludge.*—The second method of washing the sludge consists in mashing the sludge as it comes from the unwashed filter presses of the first carbonatation in a reservoir, in the bottom of which a stirrer revolves. Water is added, and when mixture is complete the whole is run into a fresh battery of filter presses. Sometimes this mashing is done in the unfiltered juice from the second carbonatation, so that the carbonate of lime which filters then more easily than that of the first carbonatation being added to the mud of the first, the cake is drier and more easy to

wash afterwards in the filter press. These mashing systems necessitate additional filter presses, and as washing in the press itself is comparatively easy, except when the beets are bad or the carbonation imperfect, the mashing process, although yielding very good results when well performed, is very seldom adopted.

*The nature of the cake a key to good or bad working.*—The filter presses are emptied by unscrewing the frames and letting the cakes of sludge fall between the feet of the framework, and then scraping the cloths. When the cake is fairly dry and well washed this is easy, and entails but little labour. But when carbonation has been badly done the mud is tacky, washes badly, and sticks to the cloths. Mere inspection of the cakes shows whether the factory is working well, or the reverse. The floor on which the filter presses are located is generally above another floor, on which the trucks of a small tramway run. In this way, by opening a trap-door between the supports of the filter press, the cakes drop into the trucks which convey them to the sludge heap, one of the necessary adjuncts to every beet-sugar factory. The trucks are emptied on to the heap thus: The floor on which the trucks run is 12 to 16 feet above ground, or more if practicable. The line of rails is prolonged outside the factory by sound staging, extending pretty far into the centre of a large courtyard. The waggons are discharged from the top of the stage, and the mud gradually forms a mound at the foot. The dimensions of the space available for this purpose ought to be calculated beforehand, so that all the sludge made during the season may be run on to it. The heap smells badly, owing to its undergoing butyric fermentation. This forms one of the nuisances of the neighbourhood of a beet-sugar factory, and should not be allowed to accumulate if at all possible. The sludge constitutes a valuable mechanical manure for certain heavy clay soils, to which it imparts lightness. But besides being a mechanical manure, this substance is also a plant-food—a manure in the true sense of the word. It contains, per cent., nitrogen, 3 to 4; phosphates, 4 to 5; organic matter, 8 to 10; lime, 25.

But sludge is very bulky and cumbersome, and it is not required in all beet-producing districts for manurial purposes. It is a long time in being got rid of, and the sugar manufacturer must wait until the autumn before the farmer wants it. An outlet for substances like this, after drying, is often to be had by selling it to the makers of chemical manures, who utilise them as driers, or make-weights for their manure. Unfortunately, beet sludge abounds in carbonate of lime, which would convert the "soluble" phosphate into "insoluble" phosphate. Its use, for this purpose, is not therefore permissible, but that does not detract from its manurial value. Moreover, the manure manufacturer can with profit send it up the cups, say 2 cwt. to the 2 ton mixing, and treat it with acid in the



usual way. If it lowered the percentage of phosphates it would in most cases at least maintain that of the nitrogen. If it be objected that it would use up a deal of acid, it must be remembered that it is precisely on his acid that the manure manufacturer makes his profit. Moreover, it should prove a corrective for those phosphatic manures which, so to speak, deliquesce after making, e.g., those made from Osso, Somme, etc., phosphates.

*Estimation of the sugar left and lost in the sludge.*—The analysis is made in duplicate by two different processes: (1) *Total sugar.*—The normal weight is triturated in a mortar with water and exactly neutralised with acetic acid, a few drops of basic acetate of lead are added, the whole made up to the mark and filtered, and polarised as far as possible in a large tube. (2) *Sugar existing as sucrales.*—The above operation is repeated without neutralisation and without precipitation with basic acetate of lead. The result gives the sugar existing as juice. Any difference between the results points to the presence of an insoluble sucrales. *Precautions.*—(a) *Sampling.*—The exact determination of the sugar lost in the sludge necessitates many precautions. The sample should correspond with the cakes of the fore, centre and aft parts of the filter press, as well as with the different parts of each cake, because the sugar is not uniformly distributed throughout the cake. A number of small samples should be taken from each filter press, and the whole mixed as uniformly as possible, so as to get a fair average sample for analysis.

(b) *Analysis.*—The sample is carefully triturated with water, and neutralised exactly with a few drops of acetic acid, verified by a drop of phenol-phthalein, otherwise there is the risk of decomposing certain compounds of lime with organic acids. These acids being dissolved by the liquid might affect the polarisation results. The amount of sugar in the sludge being small, and the solution colourless, the polarisation is made in a 500-mm. tube.

*Total weight of sludge produced annually.*—This is difficult to estimate. It is advisable to calculate it directly from the number of presses emptied, taking as a basis, from time to time, the weight of the contents of one or more presses. In default of more exact data, as the sludge contains about 25 per cent. of lime, an approximation may be made by multiplying the quicklime used in the carbonation process by 4.

*The juice from the filter presses requires further filtration to obviate the bad effects of turbid juice on subsequent operations.*—However limpid the juice as it flows from the filter presses may appear, yet it is not quite free from suspended particles, the more so if the cloths be rather loose in texture. It is very rarely that there are not some of them slightly perished, either through age or because of holes of larger or smaller size. When a filter press is set to work, the first runnings of the juice are always slightly cloudy. As all these liquids

run into the same receiver, the resultant fluid, is more or less turbid. It has already been pointed out that any sludge particles brought in the train of the juice, from the first to the second carbonatation, are so much impurities re-entering the juice owing to the fact that this sludge dissolves in the juice under the influence of the carbonic acid. Should the juices from the second stage of the double carbonatation be evaporated directly, if they be turbid, they soil and encrustate both pans and syrups. All these liquids therefore require filtration until perfectly bright and clear and altogether free from turbidity.

*Charcoal Filters.—History.*—Bone-black filtration and the chemistry of bone black will only be roughly outlined here; the subject will be more fully elaborated under Sugar Refining. In the now obsolete defecation process the juices were alkaline, owing to the presence of lime, which was sometimes used in excess. They were also rather dark in colour. The lime was subsequently injurious in the boiling, and also in the final crystallisation, and, owing to its dark colour, it was impossible to obtain fine sugar at the finish of operations. The decolorising action of charcoal on liquids containing vegetable colours was discovered somewhere about 1800. In 1811 Figuier pointed out that *animal* charcoal was a much more powerful decolorising agent than *wood* charcoal. In 1812 Derosné used bone char in refining saccharine liquids.

*The liquors were originally boiled with char, albumen, etc., from which they were separated by pressure.*—Up to 1828 finely powdered bone char was mixed with the liquid to be decolorised—juice or syrup—and it was boiled with albumen, blood, or white of egg. The albumen, in coagulating, was incorporated with the black. The whole was run into canvas bags and then pressed.

*Filtration through cylindrical tanks packed internally with bone black (Dumont filters) obviates use of albumen and renders revivification of charcoal after use practicable.*—But, as a result of the researches of Payen, Dumont constructed char filters (Fig. 41) in the shape of a cylinder open at top, of from 16 to 20 feet in height; but instead of introducing char dust he used granulated char. The juice passed through these filters was decolorised equally well as when mixed with the powder. The necessity of using albumen and bags was thus obviated; but not only so, the charcoal was left in such a condition that it was possible to revivify it. Payen found that, by reburning the char utilised in this manner, it regained its original bleaching properties.

*The revivification of bone char opens up a new epoch in the history of the sugar industry.*—Sugar factories and refineries were thereafter endowed with quite a practical method of purifying their juices, as the char could be used almost indefinitely, and the installation of Dumont's system of filtration (Fig. 41) facilitated its rational use.

Many forms of new and improved bone-char revivifying furnaces came into being.

*It constituted the first great successful step in advance which made further improvement possible.*—The use of bone char constituted such a step in advance, such a real progress, that it was owing to it that the beet-sugar industry made its first great success, and that Rousseau's carbonatation process became practicable.

*The great affinity or attraction of bone char for mineral substances.*—Bone char is also endowed with a special affinity or attraction for mineral matter, which it seizes hold of and retains in its pores when mineral solutions are passed through it. A solution of sugar, con-

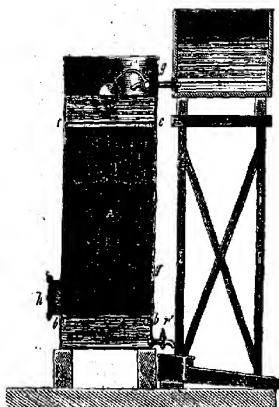


FIG. 41.—Dumont's open animal-charcoal filter.

taining a little lime filtered through granulated charcoal, issues from the filter free from lime, the latter having been absorbed by the char. The same absorption occurs with saline solutions, and a slight absorption in the case of sugar solutions.

*Bone char a purifying agent as well as a decoloriser.*—Consequently the bone-char filters not only act as decolorising agents, but also as purifying agents in the case of juices contaminated with mineral salts, and more especially organic salts. The bone char not only absorbs and retains suspended impurities, but dissolved saline substances in addition to colouring principles.

Animal-charcoal filtration was therefore regarded as a valuable aid to the older methods of working, which were not so exact as

those now in vogue. Its use, therefore, soon became quite general in all sugar factories and refineries, who used it quite regardless of cost, because by its use alone could a sufficiently clear and bright juice, prone to crystallise rapidly, and with a big yield, be obtained with certainty.

*The double carbonation process obviated the necessity for so much bone-black filtration.*—However, on the discovery of the Perier-Possoz system of chemical purification, it was found possible to lessen the enormous quantities of bone char used.

*Its action then simply mechanical.*—Moreover, in certain factories, where carbonation was well conducted, the charcoal filtration simply acted mechanically by arresting substances in suspension.

*Bone-black filtration superseded by introduction of real mechanical filters.*—Then actual mechanical filters came into use, and are now so much improved that there is no further necessity for bone char, which has almost disappeared from the beet-sugar industry. Barely a few factories still use it in countries where the carbonation process is not so well conducted as in France. Bone char will soon simply be a memory of the past. Nevertheless, its use is still continued in those refineries which make perfectly white loaf sugar. It is practically only for this purpose that it is employed nowadays.

*Manufacture of animal charcoal.*—Animal charcoal, or more familiarly "bone char" or "bone black," is made by calcining the bones of animals without access of air. Long bones, such as the tibia and the femur, are preferred. Although both the bones of the horse as well as those of the sheep are used in charcoal burning, yet cattle bones are those which are most esteemed for this purpose. Boiled butchers' bones are the best; therefore those which have been used for culinary purposes are preferred, the 10 per cent. of fat which they still contain being previously extracted by petroleum spirit. Bones, from glue factories, from which the bulk of the ossein—constituting 33 per cent. by weight of the bone—has been extracted, are very inferior and unsuitable, and should therefore be rejected. Spongy bones—the heads of the femur, for example—should also be set aside. Animal-charcoal makers, however, use a certain quantity, which they manage to pass with the others.

*The old process of burning in pots.*—These bones, previously ground, were introduced into perfectly cylindrical pots with a flat bottom, piled in a furnace, consisting of four walls without any other exit than a small air-hole, capable, moreover, of being regulated by a damper and a chimney. A small wood fire was lighted under the first pots near the air-hole. The pots, becoming heated, give off gases, which take fire. From that time forward the furnace is heated from place to place by the combustion of the gas, which escapes successively from each of the pots. When the combustion is finished the furnace and its contents are allowed to cool, and the

bones in the pot are then found retaining their original shape, but perfectly black. The black bones constitute the animal charcoal. The black bones are ground in crushers to lumps of the size of a filbert at the most. The char dust is sifted off and sold to the refineries, and nowadays to the boot-blackening manufacturers, and the large granules are used in sugar factories and refineries to pack the bone-black filters. But char dust is four times as effective as char granules (Barbet).

Owing to the presence of soluble salts, it is necessary to wash new charcoal before use; unless this were done, they would spoil the sugar liquors by dissolving therein. Accordingly, char which has been once revived is always better than new char.

*The newer process.*—Bone black is also made by distilling bones in retorts similar to those used in gas-works. This is the only rational way of making animal charcoal, the more so because at the present day the process is far from being a lucrative one; and if in the palmy days of the sugar industry the high prices obtained rendered it unnecessary to work up the residual products, it is not so now, when animal charcoal itself is a drag on the market. By distilling the bones in gas retorts, therefore, the residual products may be collected. First there is given off an aqueous ammoniacal vapour, condensing to ammoniacal liquor, wrought up into sulphate of ammonia; then, again, a considerable amount of bone tar, or bone oil, again distilled for the production of naphtha, or wrought for the manufacture of alizarine and aniline dyes, in the same way as coal tar, over which, in some instances, it possesses decided advantages in containing certain valuable colour-forming principles (*chromogenes*), either not present in coal tar or so to a far less extent. But the chemistry of bone tar is still a *terra incognita*, except in so far as Dr. Anderson's researches threw light upon the subject in the early part of last century. In the still in which the bone tar is distilled there is left a considerable residue of bone pitch, sold to varnish makers at about threepence per pound, and used in the manufacture of Brunswick black and superblack Japan varnishes. An animal-charcoal factory, therefore, which utilises its residuals to the fullest extent, is the only one which can at the present time ever hope to be a financial success. There is a loss in the conversion of bones into animal charcoal of about 30 per cent., that is to say, 15 tons of bones yield about 10 tons of char. There are two kinds of animal charcoal filters, open filters and closed filters. The open filters (Fig. 41) used in France in beet-sugar liquor decolorisation are from 10 to 16 feet high, by 30 to 40 inches wide. The side of the cylinder, almost at the bottom, is pierced by a square manhole *h*, closed by a cast-iron door resting on a rubber joint, against which it is pressed by a screw. A perforated wrought-iron plate, *b b*, forms a false bottom.

*Charging the animal-charcoal filters.*—A coarse cloth is laid over the above perforated false bottom, the manhole door is closed, and the cylinder charged from above with bone black. Another cloth is placed on the top of the black. The juice or syrup flows through a tap on to the cloth, which prevents the liquor from digging out channels in the bone black, and thus distributes it evenly all over its surface. The juice which has percolated through the char escapes from the bottom by an opening made below the perforated false bottom, and on the same diameter a screwed pipe is fixed to this opening by a nut and ascends vertically parallel with the cylinder, being bent into the form of a swan's neck at two-thirds the height of the cylinder. The juice flows gently under a slight pressure, and the filter is always full. The flow is regulated by a tap. The swan's neck moves freely round the axis of the pipe, so that the juice may flow into one of three parallel troughs, which run along the length of the row of filters, one for juice, the second for syrup, and the third for wash-water. Several filters are thus arranged in a row, according to the size of the factory, one filtering juice, another syrup, one being washed or empty whilst another is being filled. The filter is emptied by running off the last dregs of the liquid through a tap in the bottom, whilst the dirty black is removed through the manhole in the side (h Fig., R Fig.).

*The closed charcoal filters used in beet-sugar manufacture in France.*—Closed filters have the advantage over open ones of enabling filtration to be carried out under pressure. The cylinders are generally taller and narrower than open ones; like the latter, they have a false bottom, a manhole for taking out the dirty char, and a pipe through which the clarified liquor is discharged. The top of the cylinder consists of a piece of cast iron, on which is a horizontal door hermetically sealed like the one underneath; and a pipe, through which the liquid to be filtered, is run into the cylinder. The main advantage of closed filters is that they lend themselves to filtration under pressure. The filters being constantly full, an even flow is assured, without risk of overflowing, so that all that has to be done is to regulate the tap on the beak of the swan's neck so as to get the rate of flow desired, whilst in open filters both discharge and arrival taps must be adjusted simultaneously. Should the percolation through a single filter not completely purify the liquid, it may be run from the first filter to a second, and, if need be, even from the second to a third, thus doubling or even trebling the height of black through which the juice or syrup percolates.

In a well-conducted factory special filters should be allocated for the juices, whilst at the same time others are solely used for syrup. Char acts more efficiently on dense than on specifically lighter liquids. At one time manufacturers got into a deplorable habit. When a filter had been used for syrup, they ran in juice on to

the top of the syrup to force the syrup out of the filter, and that same filter was still used to filter juice as long as it came through clear. The objection to this method was that it redissolved in the juice a portion of the impurities left by the syrup owing to its great density. Analysis shows this very emphatically. Formerly chemists in sugar-beet factories were very rare. Such pernicious habits can therefore be readily understood. Bone black has now almost disappeared from beet-sugar factories, and in those exceptional cases where it is still used the whole process is subject to chemical control. As soon as the juice begins to come through turbid the process is stopped, and the contents of the filter washed with water until it does not mark more than  $1^{\circ}$  B., sp. gr. 1.0069. As the wash-water is very impure, only the first portion is sent to be treated, the remainder being used for pulping or diffusion, for washing the sludge or slaking the lime.

*Loss of sugar in the bone black and its wash-water.*—This is easily determined. The weight of bone black used and the number and capacity of the filters emptied during a given period is easily ascertained. The wash-water is about equal to half the bone black used, and the sugar left in the black is about equal to the sugar in the wash-water. All the data are therefore to hand for estimating the loss in question. But the bone black should be occasionally tested after addition of carbonate of soda. The exhausted black is collected, dried and weighed, so as to determine the ratio between the wet and the dry bone black.

*Washing and revivification of bone char.*—Bone black is revived in French sugar-beet factories by a chemical process followed by calcination in closed vessels. The char after use contains a large amount of carbonate of lime, organic salts of lime, nitrogenous and non-nitrogenous matters—in fact, soluble and insoluble salts. As it comes from the filters, it is run into capacious wooden or masonry vats, and covered with water. Hydrochloric acid is then added. The acid dissolves the lime and the organic acids, decomposes certain salts, and restores the porosity of which the black was deprived by the substances which it had absorbed. In Russia, etc., instead of using acid, a little more sugar is left in the char, so that the contents of the vats soon ferment. The carbonic acid evolved attacks the carbonate of lime, converts it into soluble bicarbonate of lime, and acts on the organic salts of lime in the same way. As soon as the alcoholic fermentation ceases the lactic sets in, and the bone char is spontaneously purified, but the stench from these fermenting vats is horribly bad; consequently, where hydrochloric acid can be got at a reasonable price it is much preferable. The bone char is taken out of the vats and washed with a bulky volume of water in a washer, on the principle of a rudimentary Archimedean screw turning in a slightly inclined trough, and causing the black to





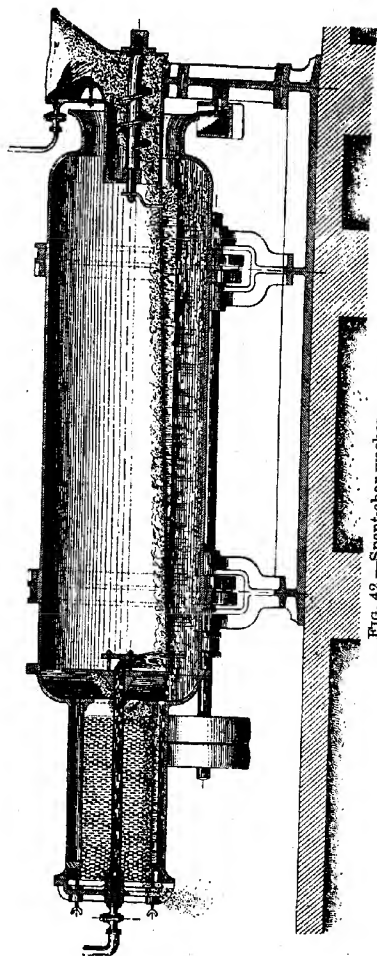


FIG. 42.—Spent-char washer.

ascend from below upwards, so as to meet a current of water flowing from above downwards. Another widely-used washer consists of a horizontal cylinder bearing obstacles in its interior and turning on its axis. The black is rolled in the cylinder in a current of water flowing in an opposite direction, and issues washed at the opposite end of the machine to where it entered. This is a better system than the preceding, as the black is not crushed so much. Again, a very ingenious washer consists of a series of hand-shaped rakes which raise the black from stage to stage in a sort of inclined

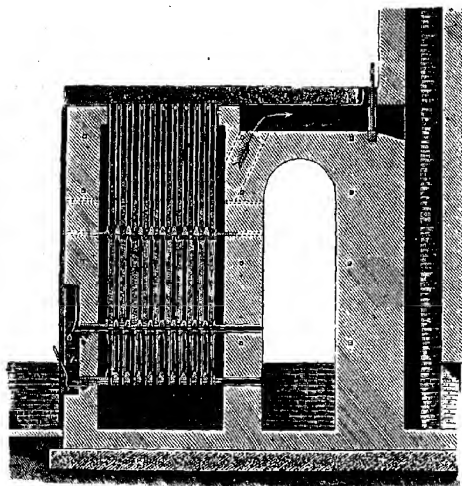


FIG. 43.—Animal-charcoal revivifying furnace.

trough, divided by partitions into a series of consecutive compartments. Whatever system of washing be adopted, in addition to char of the normal grist it yields more or less of fine char—char dust, separated by the water and sold as manure. The washed char is then steamed in cylinders to still further cleanse it. It is then transferred to the revivifying furnaces, the rôle of which is to burn off the organic matter which it contains in closed vessels.

*Revivification of bone char.*—There are a great variety of such furnaces in use. They all consist of inclined or vertical cast-iron retorts, arranged in two rows in a furnace. The head of the retort

comes out of the top of the furnace and rests on the surface of the masonry, which is covered with a large cast-iron plate, with as many openings as there are retorts. The lower end is closed by a movable, sliding or hinged door. The moist char is thrown on the cast-iron plate covering the top of the furnace. The latter being heated to such a temperature that the retorts passing through it are heated to a cherry red, the water is slowly evaporated. The dry char is now shovelled into the retorts, and when sufficiently revived the retorts are discharged into special trucks, in which it is transferred to the charcoal warehouse, to be used over again as required. The retorts are partially emptied at regular intervals by withdrawing small consecutive portions by the bottom door, and they are filled from the top in proportion as the charge is withdrawn from underneath.

*Revivified bone black denser and more compact than new bone black.*

—Revivified bone black is a little more compact than when new, because the heat to which it has been raised has caused certain more sensitive portions to fuse.

*The char cannot be revived ad infinitum.*—After being revived a certain number of times the charcoal no longer exercises the decolorising properties for which it is esteemed. It is of no further use, being non-porous. It is spent. This spent char is of no further use except for the manufacture of artificial manure, for which the high percentage of phosphoric acid pre-eminently fits it, but it makes a very dirty sooty-looking manure, to which farmers, with their inveterate prejudices, do not take to kindly. Spent char is also sold to paint manufacturers, who “fake it up” by striking a small percentage of Prussian blue on to it; or incorporate a small percentage of ultramarine blue, when it is ground. Owing to its being much harder than when new, for reasons just explained, it is much more difficult and more costly to grind than new black. Nevertheless, some paint manufacturers still grind the spent char with water in the manner described, pass it sometimes through a perforated plate, in the form of drops, and sell it as “*Superior Genuine Ivory Drop Black*”. Spent char is not fit for use in making boot blacking; the boot blacking goes mouldy and gives no lustre; indeed, for both blacking and paint purposes it would be more economical in the end to use a superior new bone black, which, if dearer at the outset, would entail far less labour and yield a far superior product. It is of little use for a paint manufacturer to try to match a paint made from new char with one made from spent char. No amount of faking will make anything but a very bad imitation. The same remark applies to blacking.

*Bone black superseded by mechanical filters.*—Such is briefly the history of animal-charcoal filtration in sugar manufactories. The methods generally yielded but little result from a purification point

of view, because enough was not used. Certain factories indeed, seeing the small effect it produced, no longer used it except as a mechanical filtration agent to arrest the suspended impurities. It was then that the real mechanical filters appeared on the scene and displaced animal charcoal.

*Early mechanical filtration.*—This consisted in the beginning in the use of a small quantity of animal charcoal, and generally of old charcoal, having nothing but its colour to indicate its origin, and equally devoid of both porosity and bleaching effect.

*Sand filters give way to Puvrez' bag filters.*—Attempts made to replace char by coarse sand did not succeed very well, and experiments with it did not last long, for Puvrez' bag filters came on the scene in 1884-85. Recent attempts with sand filters seem to have been attended with a certain amount of success.

Puvrez' bag filters, it was said, were only imitations of the old Taylor's bag filters. But Taylor's filters (Fig. 96, p. 293) never gave good results in beet-sugar factories. Puvrez, by causing the dull turbid juices to flow through the pores of the bag-shaped filter cloth *without any exterior sheath* and under feeble pressure, barely 3 to 5 feet of liquid, was able, with that simple method of filtration, to impart to these juices a limpidity unknown up to that time. Therein consisted the novelty, which made a great commotion. Puvrez' secret consisted in the careful selection of the filter cloth, a cross fabric specially designed for this purpose and answering perfectly all the requirements of a good filter cloth. His filters consisted simply of cloth pipes from 6 to 13 feet long, closed at one end and joined at the other to the shank of a tap. The bag was laid in a trough and rested on an iron grating, which kept it from touching the bottom of the trough. A series of bags were arranged side by side, each in a similar compartment, and received the juice of the filter presses collected in a shallow tank. The bags filled with juice formed a big pipe, which exuded a perfectly clear liquid from the whole of its surface. But it is needless to state that such a cloth will not filter muddy liquids, because the pores are quickly closed up. The object of such a filter is only to clarify slightly cloudy liquids. When the so-called limpid juices of the sugar factory are run into the bags they leave a certain amount of deposit, invisible in the bulk of liquid but very tangible when collected.

Puvrez thus opened up quite a new field to manufacturers, and filtration through cloth, much simpler and more economical than filtration through bone black, immediately became general. The filtered juices gave a much superior product quite equal to that from filters with insufficient bone char. But his filters were clumsy and inconveniently arranged. Their renewal was a dirty job, because when the juices failed to pass through, it was necessary to empty

them of all their contents by undoing the string which held them to the union of the tap, and this was unpleasant for the workmen, who burned their hands and threw mud in every direction. To remedy these defects a more mechanical mounting was given to the bags.

*Daneck's mechanical filters.*—The first mechanical filters fulfilling all the necessary requirements were constructed about 1887 by the firm of Breitfeld Daneck of Prague. They were invented by an engineer of that firm residing in Russia, M. Prokché, and are known as Daneck filters.

The filter consists essentially of a square wrought-iron tank 3' 3" x 3' 3" x 3' 3". The bottom consists of two surfaces inclined at 45°, and the top, closed by a hinged cast-iron door with counterpoise, makes a tight joint by an indiarubber washer, and tightening screws fixed all round it. The whole rests on four feet. The cloth bags, stretched on a corrugated sheet of iron, were arranged inside the tank. The sheets of iron with their bags were suspended, to the number of about forty, in the tanks by means of nozzles resting at each end on special supports. The nozzles were iron pipes like gas-piping, and they were enveloped by the mouth of the bag, which was folded round and fixed to them. The sheet iron penetrated into these tubes, leaving interstices which put the interior of the tubes in communication with the interior of the bags. One end of the tubular nozzle was closed, and the other open. In the top of the tank there were arranged, in two horizontal lines very close to each other, as many tubulures as there were bags suspended in the interior. The tubular nozzles penetrated into these tubulures by their open extremity by an indiarubber connection, whilst the closed extremity rested on a screw support, which fixed it firmly in its place by pressing against the tubulure. Underneath the tubulures on the outside of the tank was a trough. Its mode of action was as follows: The juice to be filtered was run into the closed tank, under a pressure of 6 to 10 feet of liquid. The juice passed from the exterior to the interior of the cloth bags, entered the tubular nozzles, traversed the tubulure and issued from the tank by running into the trough. Consequently, the deposit was made on the outside of the cloths and not in the interior, as in Puvrez' bag filters. As the bags could be packed very closely, a large filtering surface was combined in a small space, and as the tank was closed no overflow could be feared. The filtered juice being only cooled very slightly, no steam is formed on the outside, and all that had to be done as regards cleaning was to close the juice tap for running in the juice, empty the tank, open the lid, remove the corrugated sheets of iron with their bags and replace them by others. All this was effected rapidly and neatly, and presented every guarantee for the subsequent effectual cleaning of the bags. Daneck's filter

may be said to have attained perfection from the first. All accomplished since is only more or less successful imitation. One of these, however, Philippe's filter, shows some slight improvement in the mounting.

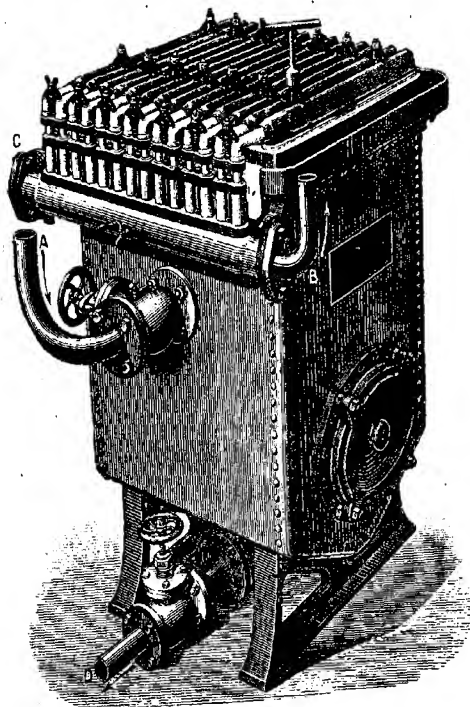


FIG. 44.—Mechanical filter (PHILIPPE).

In Philippe's filter (Fig. 44) the lid (Figs. 45A and 45B) is dispensed with, as well as its cumbersome counterpoise. The top of the tank is closed, and is pierced by a series of apertures to afford passage to the bags and their undulating iron sheet. The hollow nozzles do not enter into the tank, being longer than the apertures.

A cap is then placed over these, which covers them completely, and which bears on the front of it a tubulure for the discharge of the

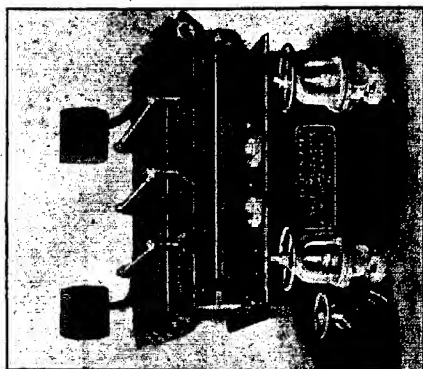


FIG. 45a.—Kazelowski's filter—closed (Cair).

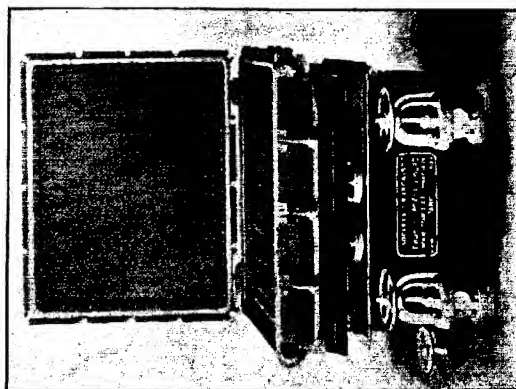


FIG. 45b.—Kazelowski's filter—open (Cair).

juice. The joint of the cap is made on the top of the tank by means of the cloth itself, which is specially arranged for the purpose, and by means of pressure screws easily undone. The renewal

of cloths only requires the lifting of the caps ; and if, for example, one of the cloths is pierced, it may be removed without interfering with the joints of the others. Consequently, Philippe's filter has some advantages over Daneck's in the handling of the framework. *Kazelowski's filter*.—Another filter, namely Kazelowski's, differs in no way from Daneck's filter except that the corrugated wrought iron is replaced by corrugated wire gauze. Certain engineering firms, nevertheless, make this filter only, but its yield is just the same. Generally each frame of each of these filters represents a square metre of filtering surface. Filters, therefore, with twenty or forty frames represent as many square metres of surface. Mechanical filtration has been applied to the juice of the first carbonatation, to that of the second, as well as to the syrups. As, owing to its alkalinity, first carbonatation juice filters with difficulty, and requires a great extent of filtering surface, this filtration is often suppressed, but wrongly so. The majority of factories have no filtration plant, except for concentrated juice before evaporation and for syrups. Mechanical filtration is a very simple and comparatively compact portion of the work, and is one of the most interesting in the purification of the juice. When well performed, it largely increases the yield of the beetroot in that grade of sugar known as "firsts".



## CHAPTER IV.

### CONCENTRATION OF BEET JUICE TO SYRUP IN MULTIPLE-EFFECT EVAPORATION VESSELS (IN VACUO).

*DIFFERENTIATION of evaporation from distillation.*—The object of evaporation is to increase the original strength of a liquid in solids from  $r_y$  per cent. to a greater strength  $r_n$  by elimination of water. In *evaporation* it is sought to recover the fixed residue; in *distillation*, recovery of the volatile ingredient is the main object, that of the fixed residue a secondary one. The amount of water to be evaporated is ascertained thus: If there be  $r_y$  lb. of solids in 100 lb. of liquid, and if the  $r_y$  lb. are to become  $r_n$  per cent. in the concentrated liquid, then the weight  $U$  of the concentrated liquid is given by—

$$r_y : U = r_n : 100, \text{ or } U = \frac{r_y 100}{r_n}.$$

Again, the weight of water to be evaporated from 100 lb. of liquid is

$$100 - U = 100 - \frac{r_y 100}{r_n} = 100 \left( 1 - \frac{r_y}{r_n} \right),$$

and the weight of water to be evaporated from  $W$  lb. of a liquid which contains  $r_y$  per cent. of solids, in order to concentrate it to the strength of  $r_n$  per cent., is

$$W - U = W \left( 1 - \frac{r_y}{r_n} \right).$$

*Example.*—1,000 lb. of juice containing 10 per cent. of solids is to be evaporated so that the residue will contain 80 per cent. of solids. Then—

$$W - U = 1000 \left( 1 - \frac{10}{80} \right) = 875.$$

The accompanying table shows the quantity of water in lb. to be evaporated from 100 lb. of juice or weak saccharine liquors containing from 1 to 20 per cent. of solid matter to so concentrate them as to bring the total solids to 80 per cent.

# CONCENTRATION OF BEET JUICE TO SYRUP 125

TABLE XXV.

A.	B.	A.	B.	A.	B.	A.	B.
1	98.75	6	92.50	11	86.25	16	80.00
2	97.50	7	91.25	12	85.00	17	78.75
3	96.25	8	90.00	13	83.75	18	77.50
4	95.00	9	88.75	14	82.50	19	76.25
5	93.75	10	87.50	15	81.25	20	75.00

A = strength of juice or saccharine liquor in solids per cent. B = lb. of water to be evaporated to bring the total solids to 80 per cent.

It may be surprising to those who have not studied the matter that, after concentrating a liquid which already contains 20 per cent. of solid matter by vaporising three-fourths of its original weight, it should even then only contain 80 per cent. of solid matter. But a little reflection will soon show the truth of the statement.

*The outlay on fuel a preponderating item of expenditure.*—It follows, therefore, that the coal bill undoubtedly forms, even at the present day, one of the largest items of the expenditure incidental to the working, carrying on, and maintenance of a beet-sugar factory. The reason for this is not far to seek. It must be borne in mind that in addition to the large amount of steam used up by the very heavy motor power required to drive the machinery, and by the continued reheating of the juice over and over again, both of which operations use up and consume much steam, there is still another and final operation which of necessity also involves a great consumption of steam and consequently of fuel. It is, in addition to the above onerous operations, absolutely necessary to completely evaporate almost the whole of the water in which the sugar is dissolved to get a syrup with a tendency to crystallise. From a historical point of view the history of the evaporation of the sugar juice may be divided into three periods: (1) The period of the open pan over a naked fire. (2) The period of the Howard or single-effect vacuum pan. (3) The multiple-effect system of the present day.

*The old-fashioned wasteful system of evaporation in open pans over a naked fire.*—The boiling down of the juice to the crystallisation-point was for a very long period, in the history of the sugar industry, carried on, with free access of air, in open pans over a naked fire. This rudimentary process has given rise to the remark that the beet-sugar factories of those days were nothing more or less than large confectionery works, where everything was carried on in as simple a style as possible, but not without leaving a considerable amount of sugar in all the residues. Needless to say, the process therefore entailed a great loss of sugar. But this was not all. There was great waste of fuel in addition to the loss of sugar. It

involved the combustion of 400 lb. of coal to obtain the paltry amount of 4 to 5 per cent. of sugar (40 to 50 lb. from 1,000 lb. of beets.) It will not, therefore, be a matter for surprise that sugar was quite beyond the reach of the poor in those countries whose supplies of sugar depended on beet sugar produced in this way. The utilisation of flue gases for heating purposes is discussed by Hausbrand, *Evaporating*, etc., pp. 12-17, where certain comparative data are given as to the working value of different fuels; and on pp. 39-55 much valuable data is given regarding heating by steam coils and steam jackets.

1828. *First use of steam in beet-sugar factories (for heating and evaporating purposes).*—A goodly portion of the nineteenth century had passed away before steam was first utilised in beet-sugar factories, but this utilisation was even then only a very partial one. It will no doubt nowadays be a matter for surprise that the first use to which steam was put in France was not to drive the machinery, but to heat the steam jackets and steam coils, which had then come into vogue for heating the defecation pans. But the industry was then only in its infancy. There were only fifty-eight factories at work in France, and thirty-one being built, whilst the area under beets was only between 9,000 and 10,000 acres.

*The introduction of steam coils paves the way.*—It was about this time that Moulfarine and Pecquer, two Paris engineers, quite independently of each other, made successful efforts to evaporate the juice by means of steam coils circulating in the liquid in the bottom of flat pans, which suit this kind of work. Moulfarine and Pecquer's innovation constituted the starting-point from which afterwards sprang the use of steam engines and heavy machinery in the beet-sugar factories of France.

*The introduction of the steam engine retarded by unfounded fears of explosion.*—The advent of the steam engine in beet-sugar factories, however, was not yet. Fear of explosions and hazy ideas as to their causes for a long time retarded its introduction, and the French were so imbued with preconceived prejudices against it that manufacturers did not want to have anything to do with it, because *steam engines were liable to explode*. It was not then so generally understood that what causes an explosion, when one does occur, in connection with a steam engine, is not the steam engine itself, but the boiler. But they already had and were actually using the boiler, the really and only dangerous element of the combination; but they did not want the engine, the harmless appliance so far as explosion is concerned.

*Howard's vacuum pan could not at first be wrought in France for want of engine to work the pump.*—The retarding influence of this ignorant prejudice will be better appreciated when it is mentioned that Howard's vacuum pan, which had been in use in Britain since about the year 1800, could not be introduced into the French factories for want of an engine to work the pump. In Howard's pan the evaporation

was effected in a vacuum, thus avoiding the enormous destruction of sugar incidental to boiling down the juice over naked fire. Howard's vacuum pan therefore gave a larger yield of superior quality sugar; but they could not be used in France because their use entailed the simultaneous employment of a steam engine to produce the vacuum. Hence, up to 1830, or even 1840, there were no vacuum pans in France. It was not, however, until railway locomotives paved the way that stationary steam engines were introduced into French beet-sugar factories.

*Derosné's and Degrand's modification of Howard's vacuum pans lowers the consumption of fuel  $37\frac{1}{2}$  per cent.*—Howard's original pan will be described in that section of this treatise which deals with sugar refining, but, as made in France, they were provided with a Degraud condenser, and were termed Derosné's machines. Their adoption in France was so satisfactory that the consumption of coal in beet-sugar factories went down from 400 lb. to 250 lb. per 1,000 lb. of beets—a saving at the very outset of  $37\frac{1}{2}$  per cent. on the costly item of fuel alone.

1852. *Introduction and adoption of the multiple-effect vacuum evaporation principle effects a further reduction of 60 per cent.*—Multiple-effect vacuum plant was first introduced into the beet-sugar industry in 1852, and this same plant, improved by its inventor in 1882, is still in general use. Rillieux thus lowered the consumption of coal to less than 100 lb. per 1,000 lb. of beets. He reduced the amount of fuel consumed by 60 per cent.

*Summary.*—The above brief epitome of the history of evaporation in beet-sugar factories is necessarily also that of the introduction of heavy machinery and plant on the large scale as they now exist, and which has converted them into engineering establishments of the first rank. But, coming back again to the technical aspect of evaporation, it will be necessary to follow it from the beginning to the present time.

*The useful effect of the heat of combustion of fuel.*—In a good boiler 1 kilogramme of coal produces about 8 kilogrammes of steam. To evaporate 8 kilogrammes of water, about 5,000 calories must be developed. Coal, in burning, produces about 7,500 calories. There is thus a loss of 2,500 calories, or a third of the heat given off by the coals. These 2,500 calories are absorbed (1) by the cold air entering beneath the grate in greater quantity than is required by theory, and escaping into the chimney as hot gases; (2) by radiation; (3) by any cause which interferes with the good working of the fires—bad stoking, boiler encrustation, bad draught, etc.

*The great loss of heat entailed in heating swing or tip-tilting pans over naked fires.*—The loss of heat is far greater beneath a pan, it being impossible with pans to apply all the improvements in the construction of a furnace that can be done with boilers; and as

swing pans were used they could not be built into the masonry. The bottom of the pan therefore was alone heated.

*The wasteful state of affairs in 1825.*—Now, as 1,000 kilogrammes of beet yield about 800 kilogrammes of juice, say 700 kilogrammes of water to be evaporated after reheating, it would be necessary (1) to burn about 40 kilogrammes of coal to defecate the juice, and (2) 280 kilogrammes to evaporate it, say 320 kilogrammes of coal for the complete treatment of the juice, rising to 400 kilogrammes, when extreme care was not taken in heating. This was the actual state of affairs in the régime of the old sugar factories in 1825.

*Improvement effected by introduction of steam.*—Matters improved, however, as soon as steam was introduced into factories; the production of the necessary heat was effected more economically, since water can be converted into steam, under much better conditions, in a steam boiler, and this steam brings its evaporative energy to bear in the steam coils of the evaporation and defecation pans with a much less perceptible loss of calories than naked fire does.

*The amount of useful heating effect of the old steam boilers.*—Steam boilers of those days barely evaporated 7 lb. of steam per lb. of coal. To heat 800 lbs. of juice to boiling-point it was necessary to condense 140 lb. of steam, requiring 20 lb. of coal. To evaporate 700 lb. of water it requires 700 lb. of steam, say 100 lb. of coal. The engines consume an amount of steam equal to about 40 lb. of coal, say, in all, about 160 lb. of coal. Radiation and considerable loss in working had also to be allowed for, because all the vessels were of copper as brilliant as possible, so that it might look well, for they did not take into account what this polishing cost, and we thus get the total, including everything, of 250 lb. of coal, on which 50 lb. at least could be economised. But they did not then go so fully into such matters. The accompanying tables embody the results of recent experiments.

TABLE XXVI.—BOILING-POINTS OF PURE AND IMPURE SOLUTIONS OF SUGAR (FRENTZEL).

A.	B.	C.	D.	A.	B.	C.	D.
	°C.	°C.	°C.		°C.	°C.	°C.
10	100·1	100·2	100·3	60	103·1	103·5	104·2
20	100·3	100·4	100·6	65	103·9	104·4	105·3
30	100·6	100·8	101·1	70	105·3	105·8	106·8
40	101·1	101·4	101·7	75	107·4	...	108·5
45	101·4	101·8	102·2	80	110·3	...	...
50	101·9	102·2	102·7	85	114·5	...	...
55	102·4	102·8	103·4				

A = degrees Balling. B, boiling-point at 760 mm. of pure sugar solutions. C, of juice and syrup. D, of low products.

*The Rillieux method.*—About 1830 Rillieux began to study the evaporation problem, which he solved by his multiple-effect system *in vacuo*. Rillieux, it appears, was born in America, but of French origin, and all his experiments were carried on in France. As early as 1830, although he was then but quite a young man, he was the best steam-engine engineer in Paris, and had already taken out patents for inventions. However, Rillieux was unable to get his invention taken up in France. In despair he tried America, but with no more success at first. In fact, it was not until about fifteen years later, namely, about 1845, that he was able to find an American manufacturer who was willing to try his apparatus on his homestead. Henceforward a number of double and triple-effect apparatus were constructed in America.

A German resident in Louisiana, however, abusing the confidence reposed in him by Rillieux, sent the plans of his apparatus to Europe, without the concurrence of the inventor. But *Tischbein* of Magdeburg, the holder of these plans, could make nothing of them. Then the firm of *Derosné & Cail* bought them from *Tischbein*, and built the first Rillieux apparatus in France which was able to work a little. By dint of groping in the dark, the Cail firm were at last able to make triple-effect apparatus which wrought almost correctly, such as were seen at the 1878 Exhibition. About this time Rillieux, who some years previously had returned to France, was struck with the poor results of the Cail triple-effect. Their plant, after all its transformations, was not even then based on the scientific principles on which it was designed by the inventor. The Cail engineers did not understand their importance and would not acknowledge that better results could be attained (see p. 140).

All treatises on "Sugar Manufacture," all works on "Heat," were full of errors in regard to evaporation by multiple-effect. Even *Horsin-Deon* acknowledges that he was quite in a maze by the constant contradictions in all such treatises.

It was about this time, *Horsin-Deon* states, that he made the acquaintance of Rillieux. Henceforward both had to struggle against routine to redress the dominant errors in the construction of such plant, but they have seen the bulk of engineering firms adopt Rillieux's ideas.

In 1882 Rillieux made further progress. He invented his multiple-effect system of heating, described further on, by which the consumption of coal is reduced sometimes below 80 kilogrammes, say 176 lb. of coal, per metric ton of beets in well-managed factories. There is a vast difference between the 400 kilogrammes of former years and the 80 kilogrammes of to-day. Moreover, it has to be noted that this difference is much greater in actual practice than appears on the face of it, seeing, when 400 kilogrammes of coal were burnt, only 800 litres of juice were extracted from 1,000

TABLE XXVII.

Results of Vaporisation of Water Experiments (Schmidt).	1892.				1893.			1896.
	I.	II.	III.		IV.	V.	VI.	VII.
Effective pressure (in kilogrammes) . . . . .	4.59	4.74	5.36		5.20	4.23	4.23	4.37
Temperature of the water (in degrees Centigrade) . . . . .	96	86.8	95		92.7	86.8	86.8	81.1
Ratio—Heating surface— Surface of grate . . . . .	39.5	52.8	53.5		45.1	79	44	73
Combustion per hour and per square metre of surface of heated surface (in kilogrammes) . . . . .	1.70	1.38	1.58		1.67	1.17	1.71	1.71
Combustion per hour and per square metre of surface of grate (in kilogrammes) . . . . .	67.4	72	84		75.5	93	74.7	136
Furnace residuum—Actual ash . . . . .	13.5	9.7	10.0		12.8	10.7	10.6	9.9
Ash of the coals by analysis per cent. . . . .	5.6	5.4	7.6		5.7	7.0	7.0	7.9
Steam produced per hour and per square metre of heated surface (in kilogrammes) . . . . .	15.9	13	14.5		15.4	11.1	15.3	15.9
Steam produced per hour and per square metre of plane of water (in kilogrammes) . . . . .	288	205	...		268	232	261	328
Steam produced per hour and per cubic metre of steam chamber (in kilogrammes) . . . . .	601	376	...		612	543	524	779
Apparent yield of steam per kilogramme of coal . . . . .	9.3	9.4	9.2		9.2	9.5	8.97	9.3
Apparent utilisation per cent. . . . .	64.8	64.2	64.5		64	69.2	65.8	67.8
100% Less in chimney . . . . .	24.4	29.3	29.4		...	23.9	27.9	17.3
Other losses per cent. . . . .	11.2	6.5	6.1		...	6.9	6.8	15.4
Price of coal per ton delivered at the factory (in francs) . . . . .	21.0	20.7	29.5		21.25	20.16	20.16	21.45
Price of 1,000 kilogrammes (metric ton) of steam (in francs) . . . . .	2.24	2.19	3.20		2.81	2.12	2.25	2.81

# CONCENTRATION OF BEET JUICE TO SYRUP 131

## XXVIII.—CONCENTRATION OF JUICE

A.	B.	C.	D.	E.	A.	B.	C.	D.	E.
1.040	7.788	686.34	87.14	16.86	1.048	6.555	554.51	84.69	20.21
1.041	7.870	665.35	86.86	17.24	1.049	6.422	541.50	84.31	20.59
1.042	7.480	647.43	86.55	17.65	1.050	6.810	530.51	84.07	20.93
1.043	7.808	629.49	86.18	18.17	1.051	6.185	517.59	83.81	21.42
1.044	7.144	613.47	85.87	18.53	1.052	6.069	505.64	83.63	21.89
1.045	6.987	597.50	85.51	18.99	1.053	5.993	494.63	83.00	22.80
1.046	6.837	582.51	85.19	19.41	1.054	5.850	484.56	82.83	22.57
1.047	6.693	568.49	84.93	19.77	1.055	5.744	478.62	82.45	23.05

A, density of extraction juice. B, hectolitres of juice required to obtain hectolitre of syrup of 25° Baumé, sp. gr. 1.211. C, kilogrammes of steam to evaporate to obtain hectolitre of syrup of 25° Baumé. D, kilogrammes of water to evaporate per hectolitre of juice to convert it into syrup of 25° Baumé. E, kilogrammes of syrup at 25° Baumé obtained per hectolitre of juice.

kilogrammes (metric ton) of beets, whilst, owing to the diffusion process, to-day 1,500 litres of juice per metric ton of beets are evaporated by 80 kilogrammes of coal. With the same quantity of 1,500 litres almost double the amount of coal would have been consumed, say, as in 1825, 750 kilogrammes of coal per metric ton of beets; and that, without the present evaporation plant, without Rillieux in fact, the diffusion process, by which a larger amount of juice is extracted from the beet, would never have been introduced into actual practice. Rillieux may, therefore, with all justice, be regarded as one of the greatest benefactors of the beet-sugar industry in particular, and of all those who profit by his system of multiple evaporation; amongst others, the British public, the largest consumers of sugar per unit of population in the world. But the improvements effected in the evaporation equipment of French beet factories have been gradual, and it is only quite recently that open-air pans have been altogether done away with.

*Howard's vacuum pans (simple-effect).*—It is necessary to anticipate matters a little and to go ahead of the course of working in a sugar-beet factory, to speak, in the first place, of simple-effect vacuum pans, which are the pans used for boiling down the syrup, the boiling operation being that which immediately follows the evaporation of the juice. But the natural order of description of evaporating vessels *in vacuo* leads us to speak first of the former. Two Paris engineers, Moullarine and Pecquer, each independently invented evaporation vessels, *flat pans heated by steam coils*, to evaporate the juice. The evaporation problem in this instance is easily solved. To evaporate a liquid it must first be brought to the boil



in order to then supply it with the necessary calories to transform it into steam.

*Definition of a calorie.*—To heat 1 kilogramme of water  $1^{\circ}\text{C}$ . it is necessary to apply to it a certain amount of heat, which is taken as unity and which is called a calorie. To heat  $n$  kilogrammes of water  $t$  degrees,  $n t$  calories of heat must be supplied. At the atmospheric pressure, if it be desired to evaporate 1 kilogramme of water, the experiments of Regnault and others have shown that, when water is at  $100^{\circ}\text{C}$ ., it requires 537 calories.

*Latent heat of steam.*—To evaporate, therefore,  $n$  kilogrammes of water it requires  $n \times 537$  calories. This number 537 is known as the latent heat of steam, which varies with the temperature of the steam formed. When steam condenses it gives up a certain number of calories. Regnault has given the formula—

$$L = 606.5 + 0.305 T.$$

$L$  being the total heat and  $T$  the temperature of the steam.

When steam condenses it does not give up its total heat, but that total heat minus the calories contained in the condensed water. As at the moment of condensation the water formed is at the same pressure, consequently at the same temperature as the steam, that is to say, at  $T$  degrees, the number of calories abandoned by the condensed steam is—

$$L = (606.5 + 0.305 T) - T = 606.5 - 0.695 T.$$

Therefore, in evaporation with free access of air by steam, it is necessary to supply the water to be evaporated with  $n(t + 537)$  calories, and to do that to condense in the coils  $x$  kilogrammes of steam yielding  $x(606.5 - 0.695 T)$ , we therefore get—

$$n(t + 537) = x(606.5 - 0.695 T).$$

*Steam calculations.*—The above formula enables us to calculate the quantity of steam required to evaporate the water in the juice. The specific heat of the juice, its boiling-point, etc., should enter into this formula, but we will not complicate it further; our object is to make the rationale of the calculations understood. The reasoning may be still further simplified: 1 kilogramme of water at  $100^{\circ}\text{C}$ . in being converted into steam absorbs 537 calories; 1 kilogramme of steam at  $100^{\circ}\text{C}$ . in condensing abandons 537 calories. It is therefore necessary to condense 1 kilogramme of steam in order to evaporate 1 kilogramme of water. That is, however, not quite exact; but, as the difference is not great, one can reason thus without being very far out. Moreover, the preceding formula says exactly the same thing, because if we take water at  $100^{\circ}\text{C}$ . we have  $t = 0$ ; with the steam at  $100^{\circ}\text{C}$ . we have—

$$n(537) = x537.$$

$$\text{Hence } n = x.$$

In evaporation calculations it is necessary to take  $t$  and  $T$  into account at each moment, as they continually vary. But in the present case, it being given that the steam used in sugar factories in the time of the open pans was low-pressure steam, and that the juice was run into them hot, the above reasoning was quite sufficient. In evaporating steam with free access of air, therefore, for every kilogramme of water evaporated it is necessary to condense 1 kilogramme of steam. For elaborate data relating to evaporation by means of steam coils and steam jackets, see Hausbrand, *Evaporating and Cooling*, pp. 39-55, to which the reader, and especially the sugar-works' engineer, is specially referred.

*The principle of Howard's vacuum pan.*—Long before Pecquer's time, Howard (about 1800) constructed and used pans for boiling sugar *in vacuo* (*vacuum pans*). This apparatus will be described fully and its principle explained under "Sugar Refining," but for purposes of illustration and comparison it is necessary to state here that these vessels consisted of steam-jacketed pans covered by a dome, in which a vacuum was made. The heating steam entered the jacket, and the steam formed by the juice, of late termed the juice steam, was led from the dome through a pipe to the condenser. The vacuum was produced by the condensation of this steam in a condenser placed at the continuation of the exit pipe, and was kept up by an *air-pump*.

*The principle of the condensation vacuum.*—The vacuum question requires explanations, generally applicable to all forms of condensers. When a vessel in which steam is generated is perfectly closed, e.g., a glass flask in a laboratory, or any kind of a boiler in a factory, if the steam filling the boiler be suddenly condensed a perfect vacuum is formed above the boiling liquid. The flattening of a tin can in which water is almost boiling by condensing the steam by sudden cooling is a familiar lecture experiment. There being a vacuum inside, the tin cannot resist the outside pressure of the air.

*The effect of gases dissolved in liquid being evaporated on the condensation vacuum.*—*Necessity for air-pump.*—It would thus only be necessary to condense the steam in a boiler to get a perfect vacuum therein. But that is not altogether correct. Boiling liquids of an organic nature, like beet juice, always contain dissolved gases or generate them. If, therefore, the steam be condensed, the gases still exist, and if they be not removed the vacuum becomes less and less perfect. There is a gradual change from the space in which there is principally steam to that in which there is chiefly air through a space in which they are mixed. Hence the necessity to have a pump as a continuation of the condenser to remove the gases. The condensers must therefore be provided with a pipe through which the air is drawn from their interior to the air-pump. This pipe must not be obstructed by water, since the variation in the pressure and amount

of air or gas introduced into the condenser would cause currents backwards and forwards so as to equalise the pressure. Water would, in fact, prevent the free extraction of the air by the pump, and thus cause irregular working.

*Capacity of pump.*—The capacity and function of air-pumps is dealt with at length by Hausbrand, *Evaporating*, etc., pp. 338-397. It is, however, necessary to give some few details at this point. This pump ought to have, in fact, a capacity proportional to the quantity of gas and air to be removed. Now, in the present instance, the condenser is a cylinder into which the steam enters, and into which a spray of cold water is injected. This cold water seizes hold of the steam, but in doing so it becomes *hot*, and this liberates all the air which it contains—and it is known that water contains  $\frac{1}{20}$  of its volume of air in solution. Consequently, when a cold-water injection condenser is used it is necessary to have a pump as a continuation of the condenser capable of removing not only (1) the gases coming from the juice itself, but also (2) the air dissolved in the condensing water. This pump, called an *air-pump*, ought to be of rather considerable proportions, the more so as the injection water represents 20 to 25 times the volume of steam to be condensed.

*The relation between the capacity of the air-pump and the volume of the water injected.*—Horsin-Deon has determined that the air-pump ought to develop per second a volume  $12\frac{1}{2}$  times greater than the volume of the water injected. This proportion corresponds to the most perfect working practically observed in the better class of condensers. Hausbrand, moreover (*Evaporation*, etc., pp. 344-376), gives elaborate information and data in regard to this important point. (See pp. 161-5.)

*The component parts of Howard's evaporation system in vacuo.*—Howard's vacuum pan, therefore, consists in principle of (1) a condensing surface, on the one side of which is the heating steam, and the other the juice to be evaporated; (2) a closed chamber in which is formed the steam from the boiling liquid; (3) an injection condenser, and (4) an air-pump.

*The useful effect of such a system.*—What will be the amount of steam required for evaporation under such conditions? It is evident, says Horsin-Deon, that it will be perceptibly the same as for a pan with free access of air. It will be a few degrees less, as the liquid boils at a slightly lower temperature, but the difference is not very appreciable. He therefore concludes that Howard's vacuum pan is of very little interest from a coal-saving point of view. But, no doubt, this is due somewhat to prejudice. Information as to the principles, advantages and disadvantages of boiling in a single-effect are given in Hausbrand's *Evaporating*, etc., pp. 56-61.

*A higher-grade sugar and increased yield obtained by lowering the*

## CONCENTRATION OF BEET JUICE TO SYRUP 185

*boiling-point.*—The essential advantage obtained, by distillation *in vacuo* is the better conditions under which the boiling syrup is placed and the better yield in sugar obtained from it. With other organic products, *e.g.*, milk, blood and albumen, they can only be satisfactorily evaporated *in vacuo*. When a vacuum exists above a liquid it boils at a lower temperature than under the atmospheric pressure, and the more perfect the vacuum the lower is the temperature in question.

*Injurious effect of hot steam on organic liquids boiled down in contact with air.*—The syrups formed by the concentration of organic juices are very easily decomposed by heat. Sugar syrups are much affected under the action of heat; the sugar caramelises and is destroyed. A beautiful straw-yellow syrup quickly changes to a black under the action of very hot steam with free access of air.

*Summary of beneficial results obtained by evaporation in vacuo.*—One of the most favourable conditions conducive to good working, therefore, is to so lower the boiling-point of the syrup that the sugar is not injured; in this way a far superior grade is obtained to that got when the syrup is boiled with free access of air. Moreover, a surplus yield is obtained consisting of all the sugar saved from destruction. That was the problem which Howard set himself to answer, and he solved it in a very fortunate manner with his vacuum pans.

*The great safety from injury to the sugar in boiling at a lower pressure than that of the atmosphere.*—As syrup boils *in vacuo* at a low temperature, it is therefore useless to heat it to the high temperature that was done in pans freely open to the atmosphere. All that has to be done is to run in steam, at a temperature a little above the boiling-point of the syrup, into the steam jacket, in order to make it boil, as is done in a well-constructed apparatus with sufficient heating surface. Moreover, if low-pressure, *i.e.*, low-temperature, steam be applied, there is much less chance of destroying the sugar, a condition highly conducive to an increased yield.

*Peculiarities and progressive alterations in the construction of Howard's vacuum pans.*—Howard's vacuum pans had an extensive enough heating surface to accomplish this, as they were very wide and held but little syrup. They met the wants of the small factories of their time. To make the capacity of the vacuum pans keep pace with the growth of the factories, the height of the pans was increased and steam coils added. Later the steam jacket was dispensed with, and the steam coils enlarged and extended. Unfortunately, the effect of the coils was miscalculated, and high-pressure steam had to be used as a source of heat. *Summary.*—Such are the vacuum pans of the present day. We shall see later on that opinions have changed and that more rational vacuum pans have been constructed for a few years back (p. 179).

According to Hausbrand (*Evaporation*, etc., p. 61), in a vacuum pan with 650 millimetres vacuum there are evaporated in one hour per square millimetre of heating surface—

By exhaust steam at 110° C.—

From water . . . . .	100 to 110 litres.
" thin liquors . . . . .	60 " 70 "
" thick liquors . . . . .	30 " 45 "

By high-pressure steam at 180° C.—

From water . . . . .	130 to 175 litres.
" thin liquors . . . . .	80 " 100 "
" thick liquors . . . . .	40 " 55 "

All that has just been enunciated, with the object of explaining the nature of *single-effect evaporation*, is a necessary preliminary to the study of *multiple-effect* plant; but the study of Howard's vacuum pan as formerly in use will be resumed under "Sugar Refining," p. 309.

*Multiple-effect evaporation plant in vacuo.—Pecquer's system.—Principles of his plant.*—Efforts were made, before Rillieux's successful attempt in 1830, to construct multiple-effect concentration systems under pressure for the economical evaporation of chemical products. These vessels were constructed on the principles of Pecquer as follows: A boiler was heated by a furnace to a *high pressure*. The pressure thus produced was used to heat a second boiler at a *lower pressure*, containing liquor coming from the first boiler. This second boiler heated a third under *still further reduced pressure*, and so on until the last, which boiled in the open air. The liquid circulated from the first into the second, and then into the third, into the fourth, and so on.

*The theory of multiple-effect without vacuum.*—The advantage of such an arrangement is easily seen. If 1 lb. of coal generates 5 lb. of steam in the first pan, these 5 lb. of steam, condensing on the heating surface of the next pan, will there evaporate other 5 lb. of steam. These second 5 lb. will again evaporate still another 5 lb., and so on, so that with four consecutive pans with 1 lb. of coal 20 lb. of water would be evaporated.

*The inherent defects of Pecquer's system.*—This method of working is known as *evaporation by multiple-effect*. But such a plan would not be at all suitable for sugar factories, because the sugar would be rapidly spoiled under the excessive pressure which such a plan would entail; for if the last pan boils at 1 atmosphere (212° F.), the one next to it would boil at 2 (250°–52 F.), the other at 3 to 4 (285°–294° F.), and the first at 5 atmospheres (309° F.). But sugar juice brought to a temperature corresponding to 5 atmospheres (309° F.) is immediately destroyed.

*Differentiation of the Rillieux system from that of Pecquer.*—Pecquer's system could not therefore be used in sugar factories. But Rillieux then invented a vacuum-evaporation system, which was a great step in advance in the sugar industry. Instead of, like

Pecquer, using as the last unit of the system a vessel boiling at atmospheric pressure, Rillieux used instead a Howard's vacuum pan which he heated by the steam coming from the juice of another pan, itself heated by low-pressure steam. He thus created a double-effect evaporation system, the second pan working in *vacuo*.

*The principle of the Rillieux system of multiple-effect evaporation in vacuo.*—The principle of such a system is as follows: The juice boiling at a very low pressure in the last pan, the jacket of this pan (supposing a Howard's vacuum pan to be used) is in contact with a liquid corresponding to the temperature of the vacuum. If this comparatively cold surface be used as a condenser for another Howard's vacuum pan, the second pan will boil under a relative vacuum corresponding to the heat of this condenser, but not so great a vacuum as that existing in the vessel used as a condenser because cold water is injected into it. There is therefore a great vacuum in the last pan and a smaller one in the one in front. Under the action of this smaller vacuum the juice boils at a rather higher temperature, but lower than if boiled at atmospheric pressure. It can therefore be heated with low-pressure steam. Rillieux used, as a source of steam for this new pan, in front of the last, another similar pan; but this latter pan he heated with virgin steam, which had no need to be of very high pressure, say half an atmosphere above the atmospheric pressure, or  $112^{\circ}$  C., say  $233^{\circ}$  F. Thus the triple-effect in *vacuo* came into existence, which the inventor, Horsin-Deon tells us, had to safeguard for fifteen years before he could find a manufacturer intelligent enough to try it in actual practice! Yet this is the system which, at the present day, constitutes the basis of all the saving in fuel hitherto effected in sugar factories.

*The modifications effected in the vacuum pan so as to convert it into a triple-effect vessel.*—In designing his triple-effect system, Rillieux made a series of alterations in the vacuum pans. Howard's pan had only a very limited heating surface; to construct an evaporating system for beet juice from similar surfaces would have been to make a child's toy. As the quantities of juice are so very bulky compared with the volume of syrup to be boiled; Howard's vacuum pans were but barely sufficient, even for the syrup itself alone. Over 80 per cent. of the juice has to be evaporated before it is made into syrup, and 50 per cent. of the syrup before it becomes crystallisable. The ratio is therefore great. See also Tables XXV., XXVIII. and XLII.

*Rillieux preferably imparted the form of a locomotive boiler to his triple-effect units.*—Rillieux, seeking a form of pan presenting a large heating surface, found no better shape to apply to his triple-effect units than the form of a locomotive boiler, which perfectly answered his purpose. His plant, therefore, was horizontal (pp. 140 and 174). The juice was in the boiler, and the steam circulated in tubes.

*Description of the various units which enter into the triple-effect*

*system.*—The triple-effect was built up thus: Three locomotive boilers were placed side by side. Steam at half-atmosphere was run into No. 1 and caused the juice in it to boil. The steam of the juice rose up into the dome of No. 1, and entered by means of a large pipe into the tubes of No. 2. The steam formed in No. 2 by the steam from No. 1 heated No. 3 in the same way, and the steam issuing from the dome of No. 3 went to the condenser and the air-pump. The function of this pump was to extract the air as well as the water which flowed into the condenser.

*Safety vessels to prevent frothing and priming.*—As the juice froths

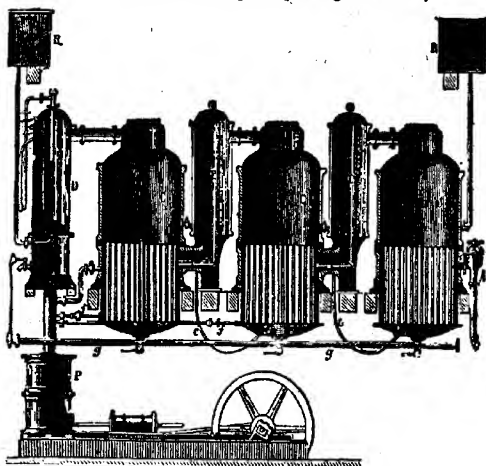


FIG. 46.—Triple-effect evaporation plant. Showing internal arrangements of No. 1, No. 2 and No. 3 vessels CCC, with their sugar recoverers TT, condenser D, and pump P (CAII).

much, there is much sugar carried over. To recover this entrained sugar, Rillieux invented safety vessels (T, Fig. 46)—boxes fixed on the course of the pipe to slacken the speed of the steam, and to allow it time to deposit the drops carried over. This vesicular carrying over of juice by the steam will be dealt with later on. To see what is going on in the evaporation vessels, Rillieux made a small aperture (Fig. 47) in the front of the boilers and inserted a pane of glass.

*Utilisation of exhaust steam from engines to heat triple-effect.*—Again, sugar factories have a certain number of steam engines, e.g., the air-pump engine. The escape steam from these engines pos-

sesses all its latent heat, and as a slight counter pressure may be imparted to the engines without injuriously affecting their working, Rillieux, instead of letting all this steam escape on to the roof, utilised it to heat No. 1, thus economising all the waste steam from the engines hitherto lost. A little direct steam from the boilers was added if the engines did not supply enough.

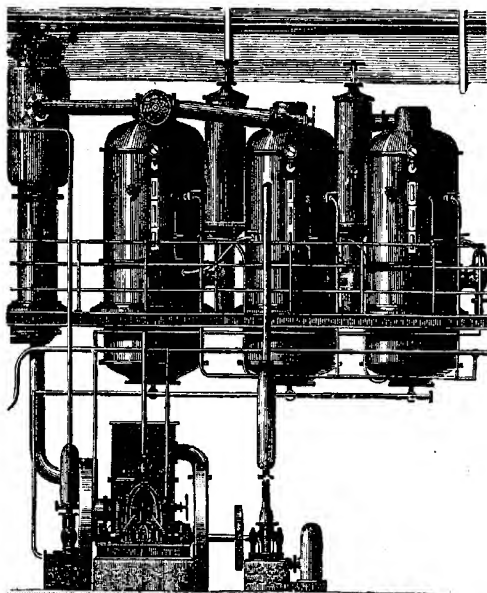


FIG. 47.—Triple-effect evaporation plant. Showing No. 1, No. 2 and No. 3 vessels with their sugar recoverers, condenser and air-pump (GAIL).

*Costly nature of the copper vessels previously in use.*—Before the adoption of Rillieux's system all evaporating pans were of copper and thus costly. Rillieux, again, was the first to substitute cast-iron and wrought-iron vessels for copper (having first ascertained that the sugar was uninjured thereby), thus greatly cheapening their prime cost.

*Change from horizontal to vertical position the only change since initiation.*—Little or no change has been effected in the original



triple-effect plant, except to change them from *horizontal* to *vertical*. In Austria, however, there would appear to be a tendency to revert to *horizontal* vessels, whilst certain German and American makers have never deviated from Rillieux's original type.

*The perfect nature of Rillieux's system demonstrated by the few improvements yet brought to bear on it.*—Rillieux's evaporation system is one which for over fifty years has not been improved upon: the only thing which differentiates the plant of the present day from that originally used is the progress made by the boiler makers in the construction of the metallic body of the plant. In Louisiana, U.S.A., where the first sets of Rillieux's plant were built, there are even now still in existence original Rillieux triple-effect plant at work, quite as well constructed as those now made, and giving the same turn-out and useful effect.

*Rillieux's triple-effect with supplementary vacuum pan misleads the European constructors of triple-effect plant.*—Rillieux, moreover, had added a vacuum "strike" pan to his triple-effect apparatus on the same plan as the evaporating vessels themselves, but the number of tubes in it was diminished by one-half. This pan was heated by No. 1 steam, so that No. 1 heated simultaneously both No. 2 and the vacuum strike pan. It was placed alongside and thus parallel with No. 3. Its condenser was common to it and the triple-effect. This was done to effect additional steam economy, but it led to unforeseen and curious results by leading astray those who adopted Rillieux's system, in Europe, without the concurrence of the inventor. The plans which Tischbein obtained consisted only of two evaporating vessels and the vacuum strike pan, and Tischbein was so ill-informed as to their nature that he took the third vessel for one of the series of evaporating vessels, and the Cail Engineering Co., who bought the plans from Tischbein, were of the same opinion. Then Cail, patenting this plant, wrote in his specification that if No. 1 was 100 metres, No. 2 should be 150, and No. 3 should be at least 200 metres. This point was strenuously debated, some writers even asserting that Rillieux knew nothing about the system which he had invented. Matters would have remained so if Rillieux had not returned, some thirty years afterwards, to France, to teach beet-sugar manufacturers the true principles of the working of the multiple-effect system, and to correct the mistakes committed by the Cail engineers, which led to a loss of 30 per cent. on the working output capacity of the plant.

*Principles of multiple-effect evaporation.—Summary of facts.*—In a multiple-effect system, No. 1 is heated by exhaust steam from engines, with aid, if need be, of virgin steam, whilst the steam from No. 1 juice heats No. 2, whilst No. 2 steam in its turn heats No. 3, the steam from which passes to the condenser and air-pump. When a vacuum is made in No. 3 there is a smaller vacuum in No.

2, and a still less in No. 1, so that, without having to use an air-pump in each vessel, by placing one at the end of the series, the vacuum is transmitted backwards in a gradually diminishing form until it reaches No. 1.

*Total fall.*—*Definition.*—Taking the steam heat to be at  $112^{\circ}$  C. ( $433^{\circ}\cdot6$  F.), and if the vacuum corresponds to a boiling temperature of  $60^{\circ}$  C. ( $140^{\circ}$  F.), the *total fall* of the temperature of the vessel will be the difference,  $112 - 60 = 52^{\circ}$  C. ( $125^{\circ}\cdot6$  F.). It is this difference between the initial and final temperature of the vessel which causes it to boil, and the greater the fall the more energetic the ebullition. All the means possible to increase this fall should be utilised collectively. Hausbrand, however, is of opinion that the amount of evaporation effected in No. 1, and also the actual consumption of heating steam, are not to any considerable extent proportional to the fall in temperature. Hausbrand also comes to the following conclusions as to the division of the heating surfaces in the most efficient manner: (1) The smallest amount of heating steam required to produce a certain amount of evaporation is used in all multiple evaporators when the fall in temperature is the same in each vessel. (2) However the fall in temperature in the separate vessels be arranged, the weight of heating steam to be supplied to No. 1 always varies within very narrow limits. Thus the manner in which the available fall in temperature is distributed amongst the separate vessels has no great influence on the economy of steam. No considerable saving in steam can be obtained by any definite division in this fall of temperature. (3) The quantity of water to be evaporated in No. 1 is on an average of the total evaporation of the multiple evaporator ( $D_1$  = total steam from No. 1 to No. 2;  $W$  = weight of juice fed into No. 1;  $U$  = weight of juice drawn from last vessel;  $d_1 d_2 d_3 d_4$  = steam from Nos. 1, 2, 3 and 4, respectively).

$$\text{In the double-effect } \frac{1}{2\cdot147} = 0\cdot466 \quad D_1 = (W - U)0\cdot466.$$

$$\text{In the triple-effect } \frac{1}{3\cdot333} = 0\cdot300 \quad D_1 = (W - U)0\cdot300.$$

$$\text{In the quadruple-effect } \frac{1}{4\cdot626} = 0\cdot216 \quad D_1 = (W - U)0\cdot216.$$

The extreme cases are—

$$\text{For the double-effect } \quad D_1 = (W - U)0\cdot434 \text{ to } 0\cdot484.$$

$$\text{For the triple-effect } \quad D_1 = (W - U)0\cdot2777 \text{ to } 0\cdot3152.$$

$$\text{For the quadruple-effect } \quad D_1 = (W - U)0\cdot1926 \text{ to } 0\cdot2335.$$

(4) The evaporation effected by heating is in all cases the least in No. 1, but the increase in the following vessels is not very great—at most 4 per cent. In the mean it may be assumed that this evaporation in the separate vessels is in the—

XXIX.—MULTIPLE-EFFECT EVAPORATION DATA (JELINEK).

	Simple-Effect.		Double-Effect.		Triple-Effect.			Quadruple-Effect.				Quintuple-Effect.				
	I.	II.	I.	II.	I.	II.	III.	I.	II.	III.	IV.	I.	II.	III.	IV.	V.
1. $t_2 - t_1$ = Difference of temperatures $\frac{52}{^\circ}$	52	26	26	26	17.8	17.3	17.3	13	13	13	13	10.4	10.4	10.4	10.4	10.4
2. $t_2$ = Temperature of the heating steam	112	112	86	112	94.6	77.4	77.4	112	99	86	78	112	101.6	91.2	80.8	70.4
3. $t_1$ = Temperature of the boiling juice	80	86	80	86	77.4	60	60	99	86	73	60	101.6	91.2	80.8	70.4	60.0
4. Indication of manometer (millimetres)	608	304	608	112	442	608	27	300	434	608	±	207	393	521	608	±
5. Absolute pressure in millimetres	152	456	152	152	648	318	152	733	460	266	152	804	553	367	239	152
6. Kilogrammes of steam condensed per hour and per square metre of surface	129.7	64.98	61.50	43.35	40.81	41.16	41.16	32.52	30.54	30.73	30.92	26.01	24.40	24.51	24.60	24.78
7. Kilogrammes of water evaporated per hour and per square metre of surface	124.8	61.50	62.28	40.81	41.16	41.52	41.52	30.54	30.73	30.92	31.11	24.40	24.51	24.60	24.73	24.74
8. Coefficient of transmission of heat—Jelinek's plant	22	22	22	22	22	22	22	22	22	22	22	22	22	22	22	22
9. Heating surface established theoretically, that of the first vessel = 100m <sup>2</sup> †	100	100	97.84	100	96.2	99	99	100	95.5	93	100	100	95.1	96.8	98.7	100.8
10. 1 Kilogramme of steam entering the first vessel evaporates kilogrammes of water	0.9	1.96	2.85	2.85	3.08	3.08	3.08	3.79	3.79	3.79	3.79	4.72	4.72	4.72	4.72	4.72
11. Kilogrammes of steam necessary to evaporate 100 of water from the juice	111.11	51.3	35.08	35.08	28.38	28.38	28.38	28.38	28.38	28.38	28.38	28.38	28.38	28.38	28.38	28.38

\* Number of vessels.

† Under pressure.

‡ The temperature of the entrance juice being = 75° C.

§ 44 millimetres above the atmospheric pressure.

# CONCENTRATION OF BEET JUICE TO SYRUP 143

Double-effect.		Triple-effect.			Quadruple-effect.			
No. 1.	No. 2.	No. 1.	No. 2.	No. 3.	No. 1.	No. 2.	No. 3.	No. 4.
$d_1$	$d_2$	$d_1$	$d_2$	$d_3$	$d_1$	$d_2$	$d_3$	$d_4$
As— 1	1.045	1	1.01	1.04	1	1.005	1.012	1.02

(5) The total quantity evaporated in the last vessel is—

In the double-effect . . . . .	0.534
In the triple-effect . . . . .	0.3703
In the quadruple-effect . . . . .	0.284
of the total evaporation of the apparatus (W - U).	

*Coefficients of transmission of heat (H. Jelinek).—*The number of calories of heat transmitted in a minute per square metre of heated surface and per degree Centigrade of difference of temperatures—

Heating the 1st carbonatation . . . . .	C = 3.47
Heating the 2nd and 3rd . . . . .	C = 7.62
Heating the filtration . . . . .	C = 7.85
To the entrance of the juice into the first vessel of the evaporating apparatus . . . . .	C = 9.65
To evaporation (Wellner-Jelinek system) . . . . .	C = 2.2
To heating the syrup . . . . .	C = 4.45

Theoretical calculation of evaporating apparatus; it being given that—

$t_0$  = Temperature of juice entering the apparatus.  
 $t_1$  = Temperature of juice boiling in the apparatus.  
 $t_2$  = Temperature of steam entering into the heating chamber.  
 $C$  = Coefficient of transmission of heat per square metre of surface in one minute, for  $t_2 - t_1 = 1^\circ \text{C}$ . This coefficient is 22 calories in the case of Wellner-Jelinek's plant, and 15 in Robert's system.

$S$  = Heating surface of the evaporating plant.

$Q$  = Quantity of heat transmitted in one minute by the heating surface of the plant, say =  $SC(t_2 - t_1)$ .

$\lambda$  = Calories necessary for transforming 1 kilogramme of water at  $0^\circ \text{C}$ . into steam of  $t_1$ , say =  $(606.5 + 0.305t_1)$ .

$r_2$  = Latent heat disengaged by the condensation of steam, say =  $(607 - 0.708t_2)$ .

$Pv$  = Weight of the condensed steam.

$Pe$  = Weight of the water evaporated by the whole surface of the evaporation plant.

Without taking into account the small losses occasioned on cooling, it will be seen when  $Q = Pvr_2$ , we get—

$$Pv = \frac{Q}{r_2};$$

$$\text{and if } Q = Pe(\lambda - t''), \text{ we get } Pe = \frac{Q}{\lambda - t''},$$

$$\text{Hence } Pe = \frac{SC(t_2 - t_1)}{606.5 + 0.305(t_1 - t_0)}.$$

To simplify calculation, we shall substitute for the evaporation of the saccharine juice that of water, and increase the heating surface of each of the vessels of the plant by 4 per cent, so as to make allowance for the different boiling-points of the two liquids.

Example I.—*Calculation of a simple-effect plant.*

Let us admit that—

$$t_0 = 75^\circ \text{ C.}$$

$$t_1 = 60^\circ \text{ C.}$$

$$t_2 = 112^\circ \text{ C.}$$

$$t_2 - t_1 = 52^\circ \text{ C.}$$

$$S = 100 \text{ square metres.}$$

$$C = 22 \text{ calories.}$$

$$P_v = \frac{114,400}{607 - 0.708 \times 112} = 216.3 \text{ kilogrammes per minute.}$$

$$P_e = \frac{114,400}{(606.5 + 0.305 \times 60) - 75} = 208 \text{ kilogrammes of water.}$$

That is, the simple-effect apparatus, having 100 square metres of heated surface, evaporates per minute 208 kilogrammes of water with an expense of 216.3 kilogrammes of steam. A square metre of surface therefore evaporates 124 kilogrammes of water per hour, and 1 kilogramme of steam evaporates  $\frac{208}{216.3} = 0.96$  kilogrammes of water.

Example II.—*Calculation of a double-effect plant.*

No. 1 Vessel—

$$t_2 - t_1 = \frac{52}{2} = 26^\circ \text{ C.}$$

$$t_2 = 112^\circ \text{ C.}$$

$$t_1 = 86^\circ \text{ C.}$$

$$t_0 = 75^\circ \text{ C.}$$

$$S = 100 \text{ square metres.}$$

$$C = 22 \text{ calories.}$$

$$r_2 = (607 - 0.708 \times 112) = 527.7.$$

$$Q = 22 \times 100 \times 26 = 57,200 \text{ calories.}$$

$$(\lambda - t_0) = (606.5 + 0.305 \times 86) - 75 = 557.73 \text{ calories.}$$

$$P_{v_1} = \frac{57,200}{527.7} = 108.3 \text{ kilogrammes of condensed steam.}$$

$$P_{e_1} = \frac{57,200}{557.73} = 102.5 \text{ kilogrammes of water evaporated.}$$

No. 2 Vessel—

$$t_2 - t_1 = \frac{52}{2} = 26^\circ \text{C.}$$

$$t_2 = 86^\circ \text{C. for No. 2 vessel.}$$

$$t_1 = 60^\circ \text{C.}$$

$$t_0 = 86^\circ \text{C.}$$

$$r_2 = (607 - 0.708 \times 86) = 546.$$

$$(\lambda - t_0) = (606.5 + 0.305 \times 60) - 86^\circ \text{C.} = 538.8.$$

$$Q = P_{v_2} r_2, \text{ or (since } P_{v_2} = P_{e_1}) = P_{e_1} r_2 = 102.5 \times 546 = 55,965.$$

$$S = \frac{55,965}{22 \times 26} = 97.84 \text{ square metres.}$$

$$P_{v_2} = P_{e_1} = 102.5 \text{ kilogrammes of condensed steam.}$$

$$P_{e_2} = \frac{55,965}{538.8} = 103.8 \text{ kilogrammes of condensed steam.}$$

A double-effect apparatus, having  $100 + 97.84$  square metres of heating surface, can evaporate per minute  $102.5 + 103.8$  kilogrammes of water. A square metre of double-effect surface will evaporate per hour only 62.5 kilogrammes of water, by expending 108.5 kilogrammes of steam on No. 1. A kilogramme of steam

will evaporate  $\frac{206}{108.5} = 1.9$  kilogramme of water. If the heating

surface of No. 2 be less than that of No. 1, it is because the juice which runs into it is of a higher temperature. Theoretically, the three vessels of a triple-effect apparatus ought each to possess the same heating surface. However, owing to the formation of encrustations on the tubes of No. 2, and to a still greater extent on those of No. 3, the surface of the latter is increased by 10 per cent. per vessel. The triple-effect is therefore so constructed that No. 1 has 150; No. 2, 165; No. 3, 185 square metres of heating surface—say 500 in all (see Table XXIX.). Take the case of a factory working 10,000 kilogrammes—say 10 metric tons—of beetroot per hour, and extracting by diffusion 14 per cent. of juice of 9° Balling (dissolved matter), sp. gr. 1.036, and making syrup of 50° Balling, sp. gr. 1.2328. The evaporating apparatus being triple-effect, the quantity of water to evaporate per hour will be—

From the diffusion juice	$\frac{10,000 \times 140}{100} \left(1 - \frac{9}{50}\right)$	. . . = 11,480 kg. (1)
„ milk of lime (= 8 per cent. CaO)	14.9 per cent.	= 1,490 kg. (2)
„ washing filter press sludge, etc.	11 „	= 1,100 kg. (3)

Say in all . . . 14,070 kg.

Say 14,100, which gives each vessel 4,700 kilogrammes—say nearly  $4\frac{1}{2}$  metric tons—of water to evaporate.

The preceding table (XXIX.) shows that in a triple-effect apparatus each square metre of surface evaporates about 40 kilogrammes of water

per hour, say, in practice, only 75 per cent. of the theoretical effect is obtained, and we get  $\frac{4700}{40 \times 0.75} =$  about 160 square metres. In the case of apparatus on Robert's system, the effect can only be taken as 50 per cent. of theory, the coefficient of transmission being only 15 instead of 22, the figure established for the Wellner-Jelinek apparatus. We thus get  $\frac{4700}{40 \times 0.50} =$  about 235 square metres.

(1) Taking  $S_1$  = dry substance (per cent. gr.) of the juice,

and  $K_1$  = its weight in kilogrammes;

\* Taking  $S_2$  = dry substance (per cent. gr.) of the syrup,

and  $K_2$  = its weight in kilogrammes;

Taking  $E$  = quantity of water to evaporate,

we get—

$$K_2 = K_1 - E, \text{ then } K_2 = \frac{K_1 S_1}{S_2}, \text{ whence } E = K_1 \left(1 - \frac{S_1}{S_2}\right).$$

(2) A litre of milk of lime of 19° B., sp. gr. 1.095, contains 193 grammes CaO, and weighs 1,152 grammes; it therefore contains 959 grammes of water to evaporate. By using 3 per cent. CaO, say 1,554 litres of milk of lime for 10,000 kilogrammes of beets, there will be 1,490 litres of water to evaporate. (3) With 8 per cent. CaO, 11 per cent. of sludge is obtained, which is washed with 100 per cent. of water, which gives 1,100 litres of water to evaporate.

*Jelinek's Austro-Hungarian data compared with Sidersky's figures for French methods of working.*—Jelinek's calculations apply chiefly to the work of Austro-Hungarian sugar factories, where generally weak juices are evaporated up to 50° Brix = 27° Baumé, sp. gr. 1.253. In France the work is very variable. Some factories extract 135 litres of juice of 4°·5 density per 100 kilogrammes of beets, others only extract 125 litres of juice with a density of 5°. In average work with beetroots of 7° density, 130 per cent. of juice with a density of 4°·7 may be relied on, say 136 kilogrammes of juice with 11·8 of dissolved substances. The quantity of water to evaporate to get a syrup of 25° Baumé (= 45°·4 Brix), sp. gr. 1.206, will therefore be  $100 \left(1 - \frac{11.8}{45.4}\right) \times 1.36 = 100.64$  kilogrammes. There is added 10 per cent. of milk of lime of 20° Baumé, sp. gr. 1.16, say 13 per cent. of the beetroots. This milk of lime contains 206 grammes CaO per litre and 956 grammes of water, say 2.64 kilogrammes per cent. CaO and 12.50 per cent. water on the beets. These 2.64 kilogrammes of CaO produce 10 kilogrammes of sludge, which is washed with double its weight, say 20 kilogrammes of water, which must be deducted 12.50 kilogrammes which return to the source and to the stirring up of the lime. The quantity of water to evaporate for 100 kilogrammes of beetroots will therefore be—

# CONCENTRATION OF BEET JUICE TO SYRUP 147

	Kilogrammes.
From the diffusion . . . . .	100.64
From the milk of lime . . . . .	12.50
From the washings of the sludge in the filter presses . . . . .	7.50
	<hr/> 120.64

This gives 12,064 kilogrammes of water to evaporate per 10,000 kilogrammes of beetroot, instead of 14,070 kilogrammes indicated by M. Jelinek. That gives 4,021 kilogrammes of water for each

vessel per hour, hence the heating surface =  $\frac{4021}{40 \times 0.75} = 134$  square

metres for the horizontal apparatus of Wellner-Jelinek, or 200 square metres for apparatus on Robert's vertical system. The heating surface required for evaporation should be 0.13 to 0.15 square metres per hectolitre of juice extracted by diffusion. The Cail Engineering Co. allows the following dimensions for its apparatus:—

	Square Metres of Heating Surface.
For 1,800 hectolitres of juice . . . . .	292
For 2,200 " . . . . .	360
For 4,000 " . . . . .	469

*Effective heat of combustion of fuel, average results from boilers in sugar manufactories.*—When the working pressure is between  $4\frac{1}{2}$  and  $5\frac{1}{2}$  kilogrammes per square centimetre, say 65-70 lb. to the square inch, and with a feed-water at a temperature between  $81^{\circ}$  and  $95^{\circ}$  C. ( $177^{\circ}$ ·8 and  $203^{\circ}$  F.) there is generally burned in a sugar works per hour per square metre (3.28 feet) of grate a quantity of coal varying between 67 and 93 kilogrammes, sometimes going beyond 100 kilogrammes. With coals giving  $5\frac{1}{2}$  to 8 per cent. of ash there is a waste in the furnaces of  $9\frac{1}{2}$  to  $13\frac{1}{2}$  per cent., and there is thus left in the cinders between 2 and 7 per cent. of combustible matter. There is lost in the smoke between 17 and 30 per cent. of the heat of the coal. The undetermined loss, that is, exterior radiation, soot and imperfect combustion represent, including the carbon in the ashes, between 6 and 10 per cent. of the heat of the coal. The remainder represents the heat utilised which, in even numbers, varies between 64 and 70 per cent. This corresponds to 9 and  $9\frac{1}{2}$  lb. of steam per lb. of coal burnt, under the above conditions of temperature of water and pressure of steam. This amount of steam represents variations between 11 and 16 kilogrammes per hour per square metre of heated surface, between 200 and 300 kilogrammes per hour and square metre of the plane of water, between 400 and 800 kilogrammes per hour and cubic metre of the steam chamber. With coals costing 20 to 21 francs, say 16s. to 17s., per metric ton delivered at the factory, the 1,000 kilogrammes, or metric ton, of steam is produced at from 2 to  $2\frac{1}{4}$  francs, say 1s. 8d. to 1s. 10d. Exceptionally, in the case of coals with heavy freight charges de-



livered at 30 francs (£1 4s.) the ton at the factory, the price of the ton of steam exceeds 3 francs (say 2s. 5d.). In 1895 about 1,000 kilogrammes of steam were consumed per ton of beetroot. This consumption in certain factories has now been diminished, and other working expenses have decreased in proportion. All steam boilers used in beet-sugar factories are good if well proportioned. The feed-waters in beet-sugar factories require the use of tubular boilers. But semi-tubular boilers may give as economical results as tubular ones of the locomotive class, by adopting heated surfaces provided with restraining bars, which practically limit the production of steam (Schmidt).

*The rational regulation of the initial and final temperatures.*—*High-pressure steam darkens the juice.*—To increase the falls, on the greatness of which the energy of ebullition depends, it might be attempted to increase the initial temperature and diminish the final temperature. But if the pressure of the initial heating steam be increased so as to raise the temperature from say  $112^{\circ}$  to  $118^{\circ}$  C. ( $233^{\circ}\cdot6$  to  $244^{\circ}\cdot4$  F.), the fall is no doubt increased, but steam at this temperature would injuriously affect the juice. The steam should be kept at a temperature of  $112^{\circ}$  C. ( $233^{\circ}\cdot6$  F.), corresponding to half-atmosphere above the ordinary atmospheric pressure, going at times as high as  $118^{\circ}$  C. ( $244^{\circ}\cdot4$  F.), but never exceeding the latter, beyond which the juice, without being spoilt so far as sugar is concerned, might be blackened by the decomposition of organic matter, unless under those special conditions of rapid circulation which are sometimes maintained in the vessels by circulators. As to the pressure in the last vessel, it can be reduced to a temperature of  $60^{\circ}$  C. ( $140^{\circ}$  F.), or  $55^{\circ}$  C. ( $131^{\circ}$  F.), or even to  $50^{\circ}$  C. ( $122^{\circ}$  F.), without any risk. The lower the temperature the better is the condition of the syrup, a result obtained by increasing the power of the condensers and air-pumps. The maker should pay most attention to this point, but he does not give it the attention it deserves.

*Horsin-Deon's formulæ and deductions.*—If we take  $m$  = the weight of the condensed steam per unit of surface and fall of temperature;  $t$  = temperature of heating steam;  $t_1$  = temperature of boiling liquid, it follows that on a surface  $S$  there will be condensed  $Q = mS(t - t_1)$ .

*Application of above formulæ.*—The quantity of steam condensed ( $m$ ) is given by the formula which we have enunciated (p. 132). To obtain a given work  $t$  and  $t_1$  depend on the installation of the system and are known. There only remains undetermined the value of  $S$ , which may be deduced from the above equation, as also can the heating surface of a system whose principal elements as given above are known. If  $t$  be the temperature of the heating steam, and  $t_1$  the temperature corresponding to the vacuum, what will be the proportional surface to give to the different vessels of

(1) a simple, (2) a double, and (3) a triple-effect? As to a simple-effect, the above formula at once gives the required surface.

*Case of equal distribution of the fall between No. 1 and No. 2 vessel.*—With a double-effect system, supposing that the total fall is spread over each of the two vessels, this distribution may be effected equally over each of the two so that the falls will be  $\frac{t-t_1}{2}$  for each vessel.

*Equal evaporative effect of No. 1 and No. 2.*—But it is known that No. 1 causing No. 2 to boil, the same amount of steam is formed in No. 1 as in No. 2, because if more steam were present in No. 1 than in No. 2 this excess could not be utilised, which is impossible; whilst if a smaller quantity were formed, No. 2 could not evaporate more than No. 1 caused it to do. Consequently, the evaporation in each of the vessels will be  $\frac{m}{2}$ . If  $S_1$  and  $S_2$  be the surfaces of the two vessels, the quantity  $Q$  of condensed steam will be—

$$Q = S_1 \times \frac{m}{2} \times \frac{t-t_1}{2} + S_2 \times \frac{m}{2} \times t-t_1 = (S_1 + S_2) \left[ \frac{m \times t-t_1}{2} \right].$$

As in the single-effect system the same amount of work is done with the surface  $S$ , we get—

$$Sm(t-t_1) = (S_1 + S_2) \frac{m(t-t_1)}{2}.$$

$$\text{Hence } 2S = S_1 + S_2.$$

*Ratio of the area of the heating surface of the single-effect system to that of the double-effect system.*—The heating surface of the double-effect system ought therefore to be double the size of the surface of the single-effect; and as  $S_1$  and  $S_2$  have the same work to do, with the same fall, they should therefore be equal, and the double effect should consist of two equal vessels, each having the same surface as that which a single-effect system would have, working in the same conditions.

*The evaporative capacity is not increased by adding more units.*—In the same way it could be demonstrated that the surface of the triple effect should be  $S + S + S$ . In a quadruple-effect system there would be four vessels equal to  $S$ , and so on. Therefore, when a vessel equal to the others of the series is added to a multiple-effect system, which by this addition is converted into a system of higher multiple-effect, the evaporating power of the system is not increased.

*The benefit lies in the less amount of steam required to heat the system, but advantages diminish progressively after the first addition.*—But the quantity of steam required to heat the system is diminished, because

with a single-effect system  $m$  kilogrammes of steam required to be applied, in the double-effect  $\frac{m}{2}$ , in the triple-effect  $\frac{m}{3}$ , then  $\frac{m}{4}$ , and so on. But as the difference between  $m$  and  $\frac{m}{2}$  is  $\frac{m}{2}$ , the difference between  $\frac{m}{3}$  and  $\frac{m}{4}$  is  $\frac{m}{12}$ , it will be seen that the passage from the single-effect to the double-effect produces six times as great an economy as the passage from the triple-effect to the quadruple-effect, and that the passage from the quadruple-effect to the quintuple-effect only effects an economy of  $\frac{m}{20}$ .

The higher the multiple the less the economy effected by another addition.—The greater the departure from the single-effect, therefore, the less is the economy obtained by adding another unit to those pre-existing; and before changing from one system to another it should be ascertained whether the resultant economy is worth the trouble of increased plant—whether, in fact, the economy realised would pay capital and interest for the new plant purchased. It has been found in actual practice that the quadruple-effect is the extreme limit to employ, the quintuple-effect not paying interest and capital on the additional vessel.

The comparative weight of steam to be condensed in single and triple-effect plants, and the effect thereof on the capacity of the air-pump.—In a single-effect plant the amount of steam to be condensed is  $m$ , in a double-effect  $\frac{m}{2}$ , in a triple-effect  $\frac{m}{3}$ , etc. Therefore, the greater

the multiple-effect the less will be the amount of condensation water necessary, and the smaller will be the air-pump required.

The variation of the "falls" from vessel to vessel in multiple-effect systems.—We have taken it for granted at the outset that, in order to ascertain the proportions to be given to the vessels of an evaporation system, that the falls were equal between each vessel. In reality, however, this is not so. The falls, far from being equal, keep on increasing from the first to the last vessel. Thus in a triple-effect, with vessels of equal size, the falls between each are  $9^\circ$ ,  $12^\circ$ ,  $18^\circ$  C.

This variation formerly attributed to faults of construction.—This fact had escaped the notice of those who attributed the differences which they had observed to some fault in construction.

The variation in pressure causes the same weight of steam to vary in volume from vessel to vessel.—The vacuum being different in each vessel, the same weight of steam occupies a different volume, and this volume increases with the vacuum from the first to the last vessel.

## CONCENTRATION OF BEET JUICE TO SYRUP 151

*The density of steam as affected by a greater or less vacuum, and influence thereof on rapidity of condensation.*—If we consider the unit of weight of steam per unit of condensing surface, the former will form a deeper layer the greater the vacuum. Consequently, a molecule of steam placed on the surface of the layer will have a greater space to travel before reaching the condensing surface, in proportion to the greater thickness of that layer, where the vacuum is more perfect. Therefore it will take longer time to traverse this distance, according to the formula  $e = \frac{1}{2} gt^2$ .

$$\text{Hence } t = \sqrt{\frac{2e}{g}}$$

If the molecule takes longer to traverse the distance which separates it from the condensing surface, the condensation will be retarded proportionately in the same time. Therefore, with the same fall of heat the last vessel will, in the same space of time, condense less steam than the first. For the plant to work properly, it is indispensable that each of the vessels condense the same amount of steam; the last vessel thus requires a greater fall of heat than the first, which may be demonstrated thus:—

Let—The heating surface of a vessel =  $S$ .

The quantity of steam condensed on that surface =  $e$ .

The fall of temperature which causes the liquid to boil =  $C$ .

The specific volume of the steam =  $V$ .

The difference of pressure which induces the motion of the steam =  $h$ .

The coefficient of evaporation special to the apparatus =  $K$ .

We get the formula—

$$e = \frac{ScK \sqrt{2gh}}{V},$$

which gives the evaporation of a vessel of which all the data are known, hence—

$$K = \frac{e}{Sc} \times \frac{V}{\sqrt{2gh}},$$

which gives  $K$  in a known evaporation plant.  $K$  is constant in all triple-effect plant, with steam at the same temperature, and varies with the temperature of the steam used.

*The use and applications of above formula.*—All the various phenomena met with in multiple-effect systems discussed up to now in this treatise may be explained by means of this formula, and the proper falls of temperature which ought to occur between each vessel calculated from it.

*Effective condensation only one item in efficient working of triple-effect systems.*—But the condensation of steam is only one of the items which affect the working of a multiple-evaporation system.

*Incidental phenomena.*—When the steam is condensed it transmits its latent heat to the condensing surface which transmits it to the boiling liquid, and this liquid finally passes into the form of steam.

*Necessity for combining the various formulæ into a single comprehensive one.*—All these successive phenomena have to be taken into account and formulated, and the whole combined in a single comprehensive one. This formula, however, is very complex, and cannot be applied to the current working of evaporation plant.

*The results arrived at.*—But it has been used to establish the relative proportions which the different vessels of a triple-effect system should bear to each other, with the result, after lengthened analysis, that all the vessels should be equal, as shown in the calculations already made, provided always that the transmission of heat through the condensing sides be the same in each vessel.

*The effect of variations in heat conductivity due to encrustations.*—For special reasons outside physics the conductivity of the metal tubes is continually varying owing to encrustation on the tubes. The juice contains calcic salts, organic and inorganic, more soluble in juice than in syrup, which, as the liquid becomes concentrated, are deposited on the tubes, in very hard layers, so that the tubes of the last vessel are covered with a thicker crust than those of the preceding vessel which itself contains more than the one in front, and so on.

*The encrustations become so dense as to stop work.*—Encrustations conduct heat badly. When the layer reaches a certain thickness, variable with the nature of the deposit, the evaporation plant will not work. It must therefore be cleaned.

*Suggested possible advantage in progressively increasing theoretical surface.*—But it is evident until this breakdown occurs the conductivity for heat is different in each vessel, getting from bad to worse from the first to the last vessel. It is therefore, they say, advisable to increase the theoretical surface of the vessels most liable to encrustation, and to make a so-called differential progression in the surfaces of each of the vessels. Thus, according to Jelinek, the three vessels of a triple-effect ought to have a progressive increase of heating surface of 10 per cent. per vessel; thus, No. 1 = 150, No. 2 = 165, and No. 3 = 185, in all 500 square metres (p. 145).

*The apparent fallacy of such a course.*—But Horsin-Deon shows this progression is quite a mistake, and even useless; evaporation goes on in spite of these changes in conductivity, only the differential progression is then effected naturally, not on the surfaces which remain constant, but on the essentially variable falls of temperature in each vessel, which follow; moreover, the physical laws corresponding to the changes in the conductivity of the condensing surfaces. There is no advantage, therefore, in constructing differential vessels.

*Uniform size of the units the most rational course to adopt.*—In all

# CONCENTRATION OF BEET JUICE TO SYRUP 153

rational multiple-evaporation systems the vessels are equal, and the falls of temperature between each vessel are differential, and regulated by the law of progression of specific volumes of vapours, and diminution in conductivity of the metal through encrustation.

*Influence of density on boiling-point.*—To these it is still necessary to add that the greater the density of the sugar liquors the higher is the temperature at which they boil (see Table XXVI.).

*Effect on the "falls".*—Consequently, the falls increase proportionately from the first to the last vessel, with the increase in the boiling temperature of the liquid evaporated. Hausbrand treats the theory of multiple-effect evaporation in a somewhat different form from Horsin-Deon, but the reader must be referred to the manual of the former on *Evaporating*, etc., pp. 62-116, for details. It must suffice here to state that, with the aid of abstruse calculations, diagrams, formulæ and tables, he solves the following question, with the result given below.

*Example.*—100 litres of liquor are to be evaporated to 10 litres in a quadruple-effect evaporator, in the elements of which the temperatures 100°, 95°, 85° and 50° C. are maintained. How much water is evaporated in each vessel? The answer given by Hausbrand is:—

	No. 1. Litres.	No. 2. Litres.	No. 3. Litres.	No. 4. Litres.
Regular evaporation	20·655	20·781	20·850	21·26
Self-evaporation	0	0·7325	1·787	3·952
Total	20·655	21·4685	22·637	25·212

89·9675 litres of water.

*Claassen's conclusions from his experiments on vertical multiple-effect apparatus.*—(1) That the coefficient of transmission, and consequently the evaporative power, with a small height of juice, is from 20 to 30 per cent. greater in No. 1 and No. 2, and from 10 to 20 per cent. greater in No. 3 vessel than with a greater height of juice. (2) With normal beets, the apparatus clean and in good working order, this coefficient remains constant for several weeks. (3) It diminishes with the density of the juice. (4) It is greater the greater the pressure of the steam. (5) It diminishes proportionately with the height of the vacuum. (6) It is lower in the last vessel than in the preceding ones, even in those which have a greater heating surface. It follows from these important considerations on the calculation of the power and dimensions of evaporating apparatus that the calculations hitherto made, with the exception of those of Horsin-Deon, are based upon the incorrect supposition that the coefficient of transmission is constant in all the vessels. Jelinek acknowledges that this supposition is false, but up to now has come to no conclusion. It is only so far exact as to determine the surface

of No. 1 vessel, and to give to it the greatest possible fall of heat. But the last vessel may be smaller than the others when it is desired to attain the maximum of power. Again, we can at 100° C. still have an evaporation, with a fall of 3° to 4° C., and, without exceeding a counter pressure of 0.5 to 0.6 atmosphere, connect a fifth and even a sixth vessel to the vertical tube apparatus.

*Necessity for uniformity in trials made with evaporation apparatus (H. Claassen).*—The numerous trials made with steam boilers are regulated by well-determined methods. But experiments made with evaporating and boiling apparatus are made without any exact basis, even although the importance of similar tests be very great. Evaporation experiments may be conducted with two very different objects in view: (1) The determination of the utilisation of the heat of the steam in the apparatus. (2) The determination of the power of the apparatus. The first point cannot very well be determined; owing to faults in construction of the apparatus, an absolutely exact value cannot be obtained. The measure of the power of an apparatus is the *coefficient of transmission*, i.e., *the quantity of heat transmitted per minute per square metre for one degree C. of difference of temperature*. Data may be obtained, by experiment, from which this coefficient may be calculated. It is necessary that the experiments be made as far as possible under conditions similar to those which will be realised in actual practice when the apparatus is at work. The points which intervene in establishing the *coefficient of transmission* are: (1) Extent of heating surface. (2) Quantity of water evaporated or of steam condensed. (3) Duration of test. (4) Temperature of heating steam and of boiling liquid.

1. *Calculation of heating surface.*—In each heating tube, surface condensation and evaporation must be considered. In vertical tube apparatus, where the liquid circulates in tubes, the surface of condensation is greater than the evaporation surface. The reciprocal takes place with horizontal apparatus and coils. As far as transmission of heat is concerned, the evaporation surface is most important, for the steam condenses on the surface of condensation the moment the temperature is lowered. The interior surface of vertical apparatus is therefore taken for calculation in testing, and the exterior surface in the case of horizontal tubes and coils. But it is better to always regard the outside surface as the heating surface, and to take the outside surface alone on account of its facilitating calculations, in the case of coils. Uniformity in testing is greatly to be desired as to what is known as heating surface. Claassen proposes to calculate the surface of a tube by multiplying the inside circumference by the length—that is to say, by the distance between the exterior surfaces and the tubular plates described on p. 157.

2. *Determination of water evaporated or condensed.*—Generally the liquid entering the apparatus is measured; the capacity of the

apparatus may thus be got either by measuring the condensed water, or by that of the liquid evaporated. The first should only be used when the steam does not contain vesicular water, or when the vessel, being heated, is served by the steam juice coming from another apparatus; or, when these two apparatus are very near, and their envelopes as well as their communication pipes are isolated by non-conducting compositions. The second method, based on the amount of condensed steam, may be applied wherever this vapour is condensed, without addition of water. The condensed water is led automatically or pumped into a measuring tank.

3. *Duration of experiment.*—If the plant be working regularly and the conditions vary but little, the test may be limited to fifteen minutes, otherwise the experiment must be prolonged.

4. *Determination of temperatures.*—The exact determination of the temperature of the heating steam and of the boiling liquid is of the greatest importance, especially when they differ but little, because then each error has a great influence on the coefficient of transmission. The temperature of the heating steam is taken as it enters the steam chamber. The condensed water from the steam chamber is always lower in temperature than the steam which has produced it, and this temperature is lower in proportion as the space traversed by the water on the heated surface is longer, and consequently the fall of temperature is greater. The temperature of the water cannot be used to calculate the fall of temperature, as is sometimes done. In exact tests, only the heat contained in the condensed water, corresponding to the temperature of the tension of the steam, should be introduced. Practically the boiling temperature at the surface is taken. In the boiling of water this temperature is the same as that calculated by the tables which give the tension of steam. But, in causing liquids containing substances in solution to boil, the temperature increases. So as to make a double control of the temperature, use is made of tables for boiling saccharine juices (see Table XXVI., p. 128, etc.).

*Definition of the fall of temperature.*—It follows from the foregoing that in practical experiments the fall of temperature should be taken as the difference between the temperature of the steam entering the steam chamber and that of the surface of the boiling syrup. This fall serves to determine the coefficient of transmission. For the determination of temperatures, accurate thermometers agreeing very exactly with one another should be used. They ought, at least, to show  $\frac{1}{3}$  of a degree, or, better still,  $\frac{1}{10}$ . As these thermometers are very costly, and not to be got everywhere, Claassen describes a simple method for protecting them. The thermometer is introduced into a tube of thin sheet copper, closed only at one end, and is filled with amyllic alcohol, by which the thermometer is surrounded. The heat is thus regularly transmitted. The graduation ought to be very visible.



The manometric determinations cannot be made with ordinary spring manometers or vacuum indicators. Mercury manometers, which the operator may construct for himself, must be used. All temperatures and pressures obtained should agree exactly with the tables of tensions of saturated steam. The barometric and thermometrical readings should be taken simultaneously and repeatedly, so as to get an exact average. Claassen gives the following scheme, for an evaporation test, during which the condensed water is measured.

*Scheme for Thermo-dynamical Testing of Evaporation Plant in Sugar Works.*

1. Heating surface (interior of tubes) in square metres . . . . .
2. Duration of the test in minutes and seconds . . . . .
3. Condensed water in litres . . . . .
4. Temperature of the condensed water, °C. . . . .
5. Weight of the condensed water . . . . .
6. Tension of the heating steam in centimetres . . . . .
7. Temperature of the heating steam calculated, °C. . . . .
8. Temperature of the heating steam measured, °C. . . . .
9. Tension of the juice steam in centimetres . . . . .
10. Temperature of the juice steam measured, °C. . . . .
11. Temperature of the juice steam calculated, °C. . . . .
12. Degree Brix of the juice in the apparatus . . . . .
13. Temperature of the entering juice, °C. . . . .
14. Temperature of the boiling juice = temperature of the  
    juice steam + the elevation of the boiling-point, °C. . . . .
15. Fall of temperature, °C. . . . .
16. Water evaporated per square metre per hour in kilo-  
    grammes . . . . .
17. Water evaporated per square metre per minute for a  
    fall of 1° in kilogrammes . . . . .
18. One kilogramme of heating steam abandons calories. . . . .
19. Coefficient of transmission . . . . .

*Construction and plan of triple-effect evaporation systems.*—It is unnecessary to speak of the now obsolete *double-effect*, which only differed from the treble by the absence of the intervening vessel. The vertical *triple-effect* system—the harmonious, artistic appearance of which is due to the well-known *Cail Engineering Company* of Paris—consists of three cylindrical vessels (Fig. 46 and Fig. 47). Each vessel consists of two truncated divisions of about equal height, joined together, the one above the other, by flanges and bolts and nuts and a tight joint between them. The lower truncated part contains the tubular heating surface (Fig. 46, p. 138). It consists of a cast or wrought-iron cylinder 5 to 5½ feet in height, with a diameter varying with the capacity of the vessel. This cylinder is

provided at top and bottom with two bronze plates, called tubular plates, pierced with holes of 50 millimetres, say 2 inches, arranged very closely to each other, and in a quincunx fashion on the same vertical as the similar-sized holes on the other, the holes of the one being placed exactly on the same vertical as those of the other, consequently opposite to each other. Into these holes tinned brass tubes, of the same length as the cylinder, are passed, and firmly fixed therein so that once fixed they do not pass through the plates. These tubes with a few cross-pieces keep the two division plates the same distance apart, and thus maintain a tight joint with the enveloping cylindrical vessel. Steam enters through a large side tube (see A, Fig. 46) connected with the cylindrical vessel, condenses on the outside of the tubes, and escapes as condensed water through a small tap in the bottom of the same vessel on the opposite side to that where it enters.

*The calandria.*—Underneath the lower cylinder known as the tubular vessel, a slightly bomb-shaped bottom is fitted and bolted so as to make a tight joint with it. Above the tubular vessel is the second main trunk already mentioned, called the *calandria*, on which is a dome-shaped lid, in the centre of which is a projection through which the steam escapes.

*The manner in which each vessel is filled with juice.*—The juice is run into the vessel through a tap in the lower part of the calandria, almost at the level of the upper tubular plate. The juice flows over the plate and falls through each hole into the tubes, and from there into the lower bottom of the vessel. When that bottom is full, the level of the juice rises in the tubes until it overflows and submerges the upper tubular plate; the flow of juice is then stopped.

*The way in which steam is applied so as to heat the juice.*—Steam is now applied through the lateral tap of the cylindrical vessel. This steam impinges on the *outside* of the pipes containing the cold juice in their interior. It there condenses, and heats the juice to the boiling-point. The juice then evolves steam, which spreads through the calandria and escapes through the top of the dome, to be thence conducted, by means of a pipe, into the tubular vessel of an identical cylinder to that just described, and to heat it.

*The calandria and its accessory arrangements.*—The calandria (Fig. 47) has several accessories. First, quite at the bottom, there is a pane of glass fixed on the vessel, so that what is going on inside may be seen. There are generally three of them, one above the other. These panes are long and narrow, and enclosed in a bronze frame. In addition to these, a round glass is placed behind, so as to light up the interior by means of a light, and thus show how the juice is boiling.

*The way in which fat is introduced to prevent frothing.*—Then comes a *butter* tap, used for introducing fatty matters (the word butter is misleading, but it is the term used) so as to beat down the froth

which is formed from time to time during boiling, when the juices are not very sound, or if the carbonation has been imperfect.

*The ammoniacal gas discharge tap.*—Finally, besides the juice tap there is a tap for the escape of ammoniacal gases, described further on, a water tap to fill the vessel and wash it, a steam valve for the same purpose, and a metallic or mercury pressure gauge.

*Sampler.*—The last vessel containing the syrup includes a *sampler*, so that a certain quantity of syrup can be extracted as the evaporation proceeds. But it is a difficult matter to extract liquid from a pan in which there is a vacuum. A small reservoir (in which the vacuum is the same as that in the pan) communicates with the space containing the syrup by means of a pipe in its top which enters the pan at a higher level than the syrup, which is thus drawn into the small vessel. When it is full, the vessel is isolated by taps. The air is allowed to enter, and another tap placed underneath opened to let the syrup run out (see also p. 176).

*Safety vessel.*—*Vesicular transference of the juice in the path of the vapour.*—When the triple-effect system is boiling, the evolved steam carries over in its train saccharine vesicles, which are more abundant the more energetic the boiling and the more concentrated the liquid, a fact well known under the title of the "Vesicular Transference of Liquids by their Vapours". The word vesicular indicates that the saccharine particle assumes the form of a small hollow sphere, a sort of bubble analogous to a soap bubble, which is formed in the midst of viscous liquids. The vesicle is also produced with pure water even, but is destroyed as soon as formed. But if the liquid be viscous, like juice, the vesicle persists longer in the steam, and is carried over by it in its train into the pipes. With concentrated syrup the vesicles are so abundant that if not guarded against a considerable loss would thus occur.

*The condensed water is thus contaminated with sugar, which injuriously affects the boilers.*<sup>1</sup>—Again, the steam containing the saccharine vesicles is condensed in the following vessel. Therefore the condensed water contains sugar, and this condensed water is used to feed the boilers; sugar, the enemy of all boilers, is thus introduced into the latter. As soon as a certain amount of sugar collects in the boiler the water blackens, forms a froth in the dome, and it is no longer possible to maintain the water-level in the boiler; the indicators "blow off," the joints disappear, the boiler cracks, and serious accidents are to be feared. The only remedy is to empty all the boilers, one after another; such a mishap means a loss of time and money. It is therefore imperative to prevent sugar from passing over with the steam into the tubular space of the next vessel. Safety vessels—the so-called sugar recuperators—are placed in the path of the steam in the triple-effect systems to catch entrained sugar.

<sup>1</sup> See Schmidt's remedy, p. 161.

*The primitive anti-frothing arrangement of the early days.*—The *Hodek*.—Formerly vesicular carrying over was unknown, but they had to contend against the copious frothing produced in the vessels, owing to imperfect defecation. The safety vessel was then called the froth-breaker, and its rôle was much simpler. But it was very inefficient, and, above all, did not arrest the saccharine vesicles. Sugar in the boilers was then a common occurrence. As methods improved, although they had no froth, sugar was still carried over. Then an Austrian called *Hodek* invented the appliance which bears his name, and paved the way for the *ralentisseur* or break, now effectively and almost exclusively employed in modern quadruple-effect systems.

The froth-breaker, a closed wide column, still to be found in obsolete plant, was intercalated between each vessel. It contained in its interior a pipe of the same diameter as the steam pipe, rising nearly up to the top of the column. The steam entered by the side, and was forced to ascend a little to find the orifice of the central tube, which it had to traverse before entering the next vessel. The steam impinging on the column *might* abandon the heavy particles such as froth, but not the light vesicular particles which it carried in its train. *Hodek* sifted or sieved the steam; he made it traverse a horizontal cylinder containing several pieces of wire gauze, on which it was bound to deposit its fine particles. The *Hodek* did its work well, but it created an obstacle to the passage of the steam, the speed of which was greatly slackened, thus diminishing the output of the evaporation plant. After this the wire gauze in the *Hodek* was dispensed with, and the cylinder made wider and a little longer, and thus the steam breaker (*ralentisseur*), properly so-called, was obtained.

The theory of the *ralentisseur* had then to be established. One could well explain that the steam in passing through a narrow into a wider tube would slacken its speed so that the heavy particles were precipitated. But on analysing this phenomenon it was seen that this fall could only be effected very imperfectly, if something else did not intervene to assist the stopping of the vesicles. The to-and-fro motion of the steam in the cylinder, as well as the resulting changes of temperature, the dislocation of the vesicles and the fall of the globules thus formed, all combine in a well-calculated and designed *ralentisseur* to effectually arrest the vesicles of sugar in the steam. *Ralentisseurs* are installed in different ways at the exit of the steam from the pans, according to the space at disposal. They are sometimes fixed horizontally, the steam entering at one end and issuing from the other, but the entry and exit may be either in the axis of the cylinder or on the side indifferently, although the axial position appears preferable, especially for the entrance of the steam. Sometimes the *ralentisseur* is vertical,

fixed on the dome of the vessel, of which it looks like the prolongation. This is a very good arrangement. Again, it is fixed vertically on the side, and resembles an ordinary *safety vessel*, but then the entrance and exit are both on the top of the cylinder, with a vertical partition between the two which divides it into two unequal portions. This kind of arrangement is not so preferable, as it causes the steam

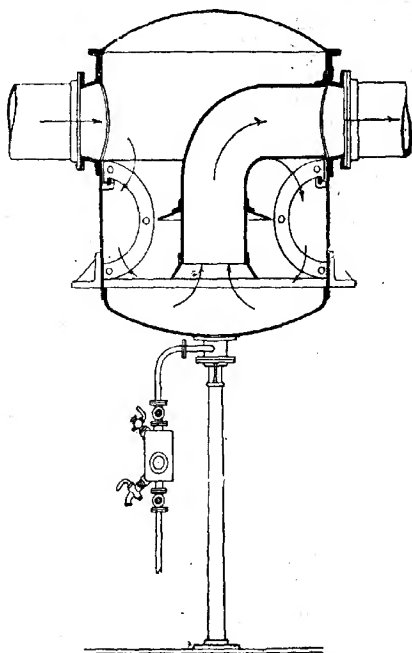


FIG. 48.—König's sugar recoverer (MAGUIN).

to make a long circuit at a sharp angle. Many other forms have been designed, but the *ralentisseur*, in all its simplicity, is the best of safety vessels. Mention may however be made of König's sugar recoverer (Fig. 48), in which the steam from the juice passes over a striated surface formed by a large number of metallic sheets, the peculiar arrangement and sections of which force the steam to follow

a very sinuous and divided path. The particles of syrup carried over are deposited along the striæ, and reunite together in each sheet, flowing by channels formed by the sheets towards a circular pipe which extracts them from the machine. In the case of the large triple-effect vessels the area of the sheets is very considerable. The apparatus is made in two general types. One type is mounted on a triple-effect vessel. In the other pattern the recoverer is placed between the steam pipes, as shown in Fig. 48. Hausbrand (in *Evaporating*, etc., pp. 117-160) treats the whole subject of the vesicular carrying over of liquids by their vapours and similar phenomena in a very exhaustive manner, but the mathematical treatment of the subject is too intricate and involved for further discussion here.

Schmidt claims to counteract the *corrosive* action of sugar in boilers by maintaining the feed-water slightly alkaline (tested after dilution with its own bulk of water and boiling) by addition of soda ash.

*Wilkowicz's radiating heater.*—The application of radiating heaters to the *horizontal* vessels of the triple-effect system, whilst appreciably increasing the heating surface, also gives rise to an energetic circulation of the juice which did not exist previously, and increases their evaporative power by more than 30 per cent. In *vertical* vessels several applications have been made of four radiating heaters placed under the lower tubular plate of the first vessel to increase its power and to allow of juice steam being raised from it for heating. An evaporation vessel or juice boiler with radiating heaters, placed in front of No. 1 and heated by direct steam, furnishes heating steam and feeds No. 1 and replaces the circulators. It may also serve as a No. 1 for an ordinary quadruple-effect, or as juice boiler before a quadruple-effect.

*Condensers and air-pumps.*—*Their place and function in the triple-effect.*—The triple-effect consists, as just seen, of three similar vessels—each provided with its sugar recoverer: No. 1 steam-heating No. 2 liquid, No. 2 steam-heating No. 3, whilst the steam from No. 3 passes to the *condenser* and the *air-pump*. Two different methods of condensation may be adopted to produce the vacuum: (1) The production of a vacuum by the so-called *wet* system of condensation; (2) By the so-called *dry* system of condensation. 1. By this method (that generally adopted) the course pursued is the same as that used for direct condensation steam engines. The air-pump extracts the condensed steam and injected water along with any uncondensed gas. The principle of opposite currents between steam and cooling water may be utilised in these condensers, but is not of great service. Wet condensers are generally arranged for parallel currents of steam and cooling water. Opposite currents may also be arranged in a wet condenser by admitting the steam below and exhausting the air above, so that the air being last in

contact with cold water may be removed colder, which is an advantage in itself. But the air in the pump cylinder, or even earlier, is in contact with the warm water, above which is steam of corresponding pressure. Thus any advantage of this construction can hardly be recognised, for the air is ultimately mixed with the water,

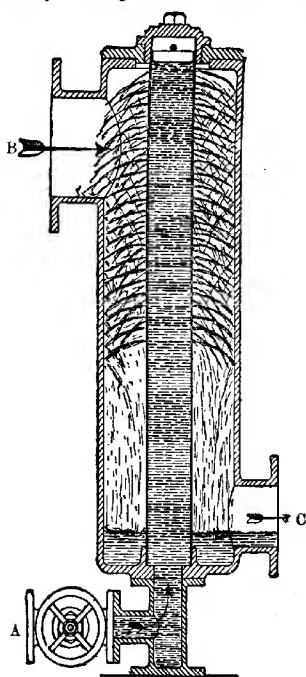


FIG. 49.—Reverse-current condenser. A, water injection valve; B, inlet for steam to be condensed; C, to air-pump.

and very rapidly acquires its temperature, when the condition of things is the same as if air and water were exhausted by the same passage (Hausbrand). The pressure in the wet air-pump is always dependent on the temperature of the water pumped out. A wet condenser with counter current (Fig. 49) consists of a vertical cylinder forming the continuation of the steam pipe from No. 3 vessel of the triple, No. 4 of the quadruple, or No. 5 of the quintuple system. Cold water is injected *underneath* by means of a rose (Fig. 46), a cone, or a vertical pipe pierced with holes, and reaching to the top of the condenser (Fig. 49), or, finally, by any kind of arrangement which shoots the water in the form of finely divided spray in an inverse direction to the steam. A side tube C at the bottom connects the condenser with the air-pump. Now air-pumps, not having a variable volume, are sometimes drowned with condensed water when a large quantity is injected, and the air can no longer be aspirated to such an extent that the vacuum is greatly affected. Attempts have therefore been made to separate the evacuation of the water from the removal of the air by *e.g.* the water barometrical column (Fig. 50).

*Air-pump.*—The air-pump is a large horizontal cylinder in which

a piston is driven by a steam engine. Above the cylinder is a chamber with three compartments containing aspiration and pressure flap valves. The compartment in the middle communicates directly with the condenser, and its two side faces, inclined at  $45^\circ$ , are provided with aspiration valves.

The two extreme compartments communicate by apertures with the two extremities of the cylinder, and are furnished on their upper face, forming a horizontal partition, with pressure valves. On this chamber is fixed a tank with overflow pipe to let the water flow away.

At each stroke of the piston the aspiration which it produces causes the air of the condenser confined in the middle compartment, and the air which is in the condenser, to enter into the compartment communicating with it, and behind it, through the apertures in the cylinder. At the return stroke this air is pressed out through the valves into the upper tank, from which the water runs away and the air escapes into the atmosphere. The water which is left after this back movement of the piston overflows all the bad spaces, and when it starts again no trace of steam or air exists which might militate against the useful effect of the pump.

*The great defect of too small pumps.*—The pump ought to be sufficiently large to aspirate the water and the large amount of gas, coming not only from the juice but also from the condensation water itself, which is heated in contact with the steam. One of the great defects of the triple-effect system is the use of pumps that are too small.

2. *The fall pipe or barometrical condenser.*—It is well known that an absolute vacuum corresponds to a column of water 33·8 feet high. If, therefore, a 33·8 feet pipe be placed under the condenser, dipping into a tank of water, the water will rise in the pipe to a height corresponding with the vacuum, and will not rise any higher. Advantage has been taken of this fact, and the condenser has been placed 33·8 feet in the air, with a pipe running down to the ground floor of the factory, and dipping into a tank with overflow pipe. When the cold water is injected into the condenser this water flows down into the pipe and escapes through the tank underneath. The requirements as to the condenser are, that it be large enough and that water can be injected into it in abundance and in a fine spray, so as to seize the steam immediately on its arrival. The barometric column with dry air-pump form of condenser is that which is mostly used in Britain, Austria and Russia. It is also beginning to be adopted both in France and Germany. The object of this form of condenser is to relieve the air-pump of having to remove the condensed water as well as the air. The amount of water used in the condenser varies with the season of the year, with, in fact, the temperature of the water to be used in condensation. The latter being colder in winter than in summer, less condensing water is required. Only, the shape



of the condenser is slightly altered so that the air which it contains may be extracted from above. It still remains a vertical cylinder, but the steam enters on the side generally from below, whilst the air is aspirated through a pipe fixed on the side of the top of the cylinder (Fig. 50).

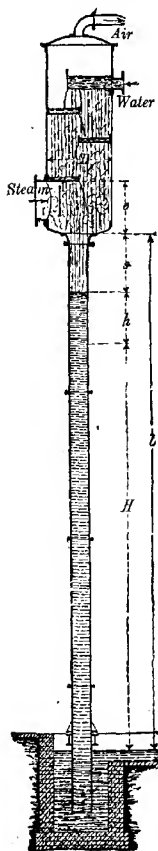


FIG. 50.—Parallel-current barometrical condenser.

valve, which causes the

*The water spray.*—The mechanical division of the water is obtained by numerous artifices constituting a series of cascades which the air has to pierce before it gets to the top. During its impeded upward progress it is thus deprived of all contained steam. Sometimes, however, the water and the steam enter at the top, and the air is extracted from the bottom of the cylinder. But generally the arrangement first described is preferred. Hausbrand remarks that the principle of opposite or counter currents is almost always applied to this class, and with great effect, so that they are also called dry counter-current condensers.

*Parallel and counter-current condensers.*—In certain factories two condensers are placed side by side, the one with *parallel-current* of water and steam (Fig. 50), the other with *counter-current* (Fig. 49) (these are the terms applied to the two types of condensers), so that the condensation is as perfect as possible and the air cooled to its maximum.

*Dry air-pumps and wet air-pumps.*—No water is extracted by the air-pump in above instance, hence it is called the *dry air-pump* in contrast with the other system, called the *wet air-pump*. These pumps ought therefore to be very perfect; bad spaces must be avoided. They are real pneumatic machines. Thus the valves are frequently replaced by drawers or by cylinders, as in the distribution of the *Corliss* engines. Moreover, an arrangement is also in use which recalls Babinet's valve, which causes the

at the end of its course, to communicate momentarily with the other portion where the vacuum is at a maximum, so as to remove any air remaining in the inevitable bad spaces. Such pumps when well constructed often make a better vacuum than those of the first system. They are smaller and less cumbersome. The defect of the system is they involve a force-pump to pump all the cold condensing water into a tank placed 36 feet up in the air, whilst the other system, being on the ground level, could draw its own water. Each system has its drawbacks.

*The pressure in the condenser.*—The pressure in the condenser is the sum of the pressure of the air, gas and steam (pp. 131-4). Warm water, which has previously been used for condensing, still contains some air, if it be only a small amount. In closed vessels partially filled with hot water, in which a considerable pressure is produced by artificial means, the water would still evolve steam of a pressure corresponding to its temperature, which would correspondingly increase the previously existing pressure. The function of the air-pumps, therefore, is to withdraw as quickly as may be the air and gas introduced by steam and juice, so that the pressure in the condenser may only consist of that due to the pressure of the steam, which depends on the temperature of the water. The pressure should be as low as possible, for as it decreases the boiling-point falls proportionately, and the useful evaporating capacity of the heating surface increases in a similar ratio. As is pointed out in the "Sugar Refining" section, in the description of the pump of Howard's vacuum pan, there can be no question of exhausting by the air-pump the steam vapour formed from the juice, together with the air, since the volume of this vapour is so great that it cannot be withdrawn by pumps of rational dimensions. If it were tried to exhaust steam from the condenser by the air-pump to induce the formation of fresh steam from the water, and cool the warm water and so produce a greater vacuum, the air-pump would have to be of quite impossible dimensions. Hausbrand (chapter xx. of his *Evaporating*, etc., p. 209) illustrates the above principles by the following example. (The reader is referred to this section of Hausbrand's treatise for much valuable information, for which no space can be found here.)

*Example.*—To condense 100 kilogrammes of steam 3,000 kilogrammes of water are required, which become heated from 15° to 35° C. To cool these 3,000 kilogrammes of water 5° C., i.e., to 30°, it would be necessary to deprive them of 15,150 calories, i.e., to evaporate  $\frac{15,150}{580} = 26.1$  kilogrammes. Now 1 kilogramme of steam at 30° to 35° C. has a volume on the average of 28,750 litres, thus 26.1 kilogrammes measure 750,375 litres. Such great volumes can naturally not be pumped out in a short time. The operation is therefore restricted to removing the air alone as quickly as possible.

*Quadruple-effect evaporation plant, with multiple-heating effects, Rillieux system.*—Quadruple-effect systems only differ from the triple-effect by an additional vessel between the first and the last vessel. Horsin-Deon constructed a quintuple-effect plant comprising five similar vessels. Cail desired to make a nonuple-effect, which would have likewise worked satisfactorily if the principles on which multiple-effect plant should be constructed had then been understood. Nothing limits the number of multiple-effects except the cost, which beyond the quadruple or quintuple-effect, with the

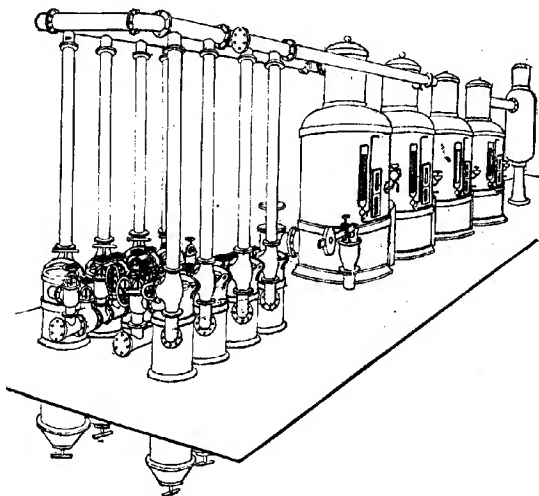


FIG. 51.—The quadruple-effect system of multiple-effect evaporation, and systematic utilisation of the steam from the vessels composing the series to reheat other juices.

complete Rillieux system, may be too dear for the economy which it effects. But with the usual complement of engines in a factory the triple-effect is the only one possible, because, as soon as the quadruple-effect, which consumes less steam than the triple-effect, is started, there is an excess of waste steam lost on the roof. It is useless to get a fourth vessel when three suffice, unless the steam be utilised as in the new Rillieux system, the principle of which is to regard each vessel of the quadruple-effect as a steam boiler, capable, if of the right size, to furnish all the steam required

to heat the next vessel, as well as for the reheating of the juice wherever necessary. We have hitherto studied only multiple-effect

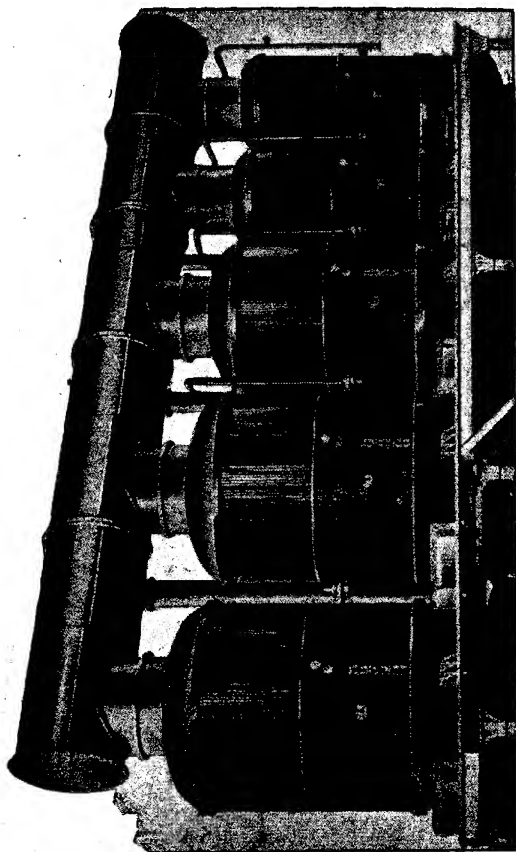


Fig. 52.—Quintuple-effect system.

evaporators in which the steam produced in No. 1 is used to heat No. 2, the same operation being repeated on No. 3 with the steam

from No. 2 without interfering with the steam in any way whatever, either by taking from it or by supplementing it. In the new system, partially spent steam is frequently taken from Nos. 1, 2, etc., for reheating the juices, thus re-utilising steam which has already removed several times its own weight of water in the multiple-effect.

*The quantity of steam required and the rational utilisation thereof in the working of a quadruple-effect system, and general factory work.* No. 1 vessel.—In the quadruple-effect No. 1 yields steam at 106° to 108° C. (222°·8 to 226°·4 F.), which heats No. 2, and also all the vessels in which it is necessary to bring the juice to 100° C. (212° F.), as in the second carbonatation, boiling the syrups to strike point, etc.

No. 2 vessel.—*The rational utilisation of its steam.*—No. 2 likewise furnishes steam at about 100° C. (212° F.). This steam, besides heating No. 3, is used to heat all those vessels where it is necessary to bring the temperature of the juice to at least 90° C. (194° F.), as in the first carbonatation, in the calorisors of the diffusion batteries, the coils in the top of the strike pan, and so on.

*The great strain upon the supply of steam in No. 1.*—To do this it will be sufficient to calculate the quantity of heat which each of the vessels ought to furnish, and to impart to each the heating surface by which they can evaporate that amount of water. No. 1 having to furnish steam for No. 2, independent of that which it will itself have to produce, will require greater heating surface than any of the others, and will generate all the steam necessary for all the heating required to be done in the factory beyond that required for the quadruple-effect.

*Assisted not only by waste steam from engines, but also by virgin steam from boilers.*—In addition to the exhaust steam it must therefore be fed with the direct steam required for the rather considerable amount of work it has to do. It would be best to take all the steam required for the various extraneous purposes from the final vessel of the series, i.e., from No. 3 of the triple, No. 4 of the quadruple and No. 5 of the quintuple-effect, but as the temperature of the steam falls very decidedly from No. 1 to No. 4 or No. 5, the steam must be taken from the vessel with that temperature which approaches most closely that of the work to be done. Hausbrand gives the saving per 100 kilogrammes of steam withdrawn as follows:—

TABLE XXX.

	Double-Effect.	Triple-Effect.	Quadruple-Effect.
No. 1 . . .	47·5	31	22·5 kilos. of heating steam.
No. 2 . . .	...	62	45·0   "       "
No. 3 . . .	...	...	67·5   "       "

## CONCENTRATION OF BEET JUICE TO SYRUP 169

*Water evaporated in each vessel when steam is taken from the individual vessels.*—The production of the steam withdrawn may be regarded as quite distinct in all the vessels from the ordinary evaporation of the liquid, and it may be taken that there are separately introduced into No. 1: (1) The water in the form of juice which is to be converted into steam in the various vessels, then to emerge partly as steam, partly as condensed water. (2) The liquor, which was originally mixed with this water but is now separate from it, and which, although it still contains the same quantity of solid matter as before, has that solid matter mixed with less water by the amount to be drawn off as steam for factory heating purposes. The liquor is therefore more concentrated than in the beginning. We can find the quantity of water to be evaporated in each vessel individually, and in the whole collectively, for the purpose of producing steam for factory-heating purposes. By subtracting this weight of water from the total weight of liquor we get the weight of liquor to be evaporated and condensed in the ordinary way. With this system, therefore, there is no fear of wasting exhaust steam, since that yielded by the engines is insufficient even if the quintuple-effect be used.

*The economy thus effected in the use of steam.*—What will be the steam economy realised under such circumstances? The reply is easy without calculations. No. 1 yields steam from the juice for all the reheatings. To evaporate this juice it will require a weight of steam about equal to that which it produces. The various heatings are therefore (1) effected by the same weight of steam, and that whether this steam be employed directly in the coils or indirectly in the multiple-effect system. But in using it in the multiple-effect a result is obtained which is not produced by sending the steam directly through the coils, and this result is the evaporation of the juice. The economy of the system therefore consists in the evaporation of the juice with the steam, formerly employed directly in the coils of the factory. This evaporation is therefore effected, so to speak, gratuitously, because it serves as the intermediary, almost without loss, to the use of direct steam. Moreover, the heating effected by the steam from No. 2 is produced by the steam coming from No. 1 so that 1 lb. of steam evaporates 2 lb. of juice before being drawn from No. 2 for reheating the juice in other stages of treatment. The economy is thus double that obtained by taking steam from No. 1 for the purposes mentioned.

If the steam to be used in diffusion, carbonatation, heating of the syrup, say in all 29 kilogrammes, has at the outset evaporated 29 kilogrammes of water out of the 80 which must be eliminated, and the evaporation instead of 30 is now only 20, 10 kilogrammes of steam are saved per hectolitre of juice, without going beyond the triple-effect and heating by No. 1; say 10 kilogrammes per 77, or

an economy of about 13 per cent. If the triple be replaced by the quadruple-effect, and if the steam used for reheating be drawn from Nos. 1 and 2, *heating by the double- and triple-effect*, the steam consumption is reduced to 54 kilogrammes per hectolitre, instead of 77, say an economy of 30 per cent. An economy of 30 per cent, may thus be effected in the steam used in sugar factories equipped with triple-effect plant. Instead of burning 120 kilogrammes (264 lb.) of coal per metric ton of beetroots, only 84 kilogrammes (185 lb.) are consumed.

TABLE XXXI. — DISTRIBUTION OF THE STEAM CONSUMPTION IN A SUGAR FACTORY PER HECTOLITRE OF JUICE (SEE ALSO TABLE XII., P. 200).

Operation.	Kilogrammes of Steam per Hectolitre of Juice, or lb. per 10 Gallons.
1. Diffusion . . . . .	8
2. First carbonatation . . . . .	14
3. Second carbonatation . . . . .	6
4. Evaporation (triple-effect) . . . . .	30
5. Heating of the syrup . . . . .	1
6. Boiling (1st and 2nd) . . . . .	8
7. Turbines and loss . . . . .	10
	...
	77

*The fuel consumption in Austrian factories, where Rillieux's system has been made a fine art, is reduced to a minimum.*—That is what has made Rillieux's process such a success—a success accentuated daily; for, in Austria, where it has been equipped in altogether favourable conditions, by utilising all the multiple-effect heatings possible in reheating, boiling (p. 179), filling, etc., they have reduced the fuel consumption to about 60 kilogrammes (132 lb.) per ton of beetroot.

*The coal bill, the chief item of expenditure besides the beet, reduced to a minimum.*—The Rillieux process is the process of the future, and every sugar factory which adopts it, in its entirety, is assured of the minimum consumption of coal. Coal, after beets, being the only serious expense in the factory, great profit accrues from lowering coal consumption to a minimum.

*The practical working of the quadruple-effect.*—The multiple-effect heating, i.e., heating by the repeated re-use of steam, in conjunction with the quadruple-effect is executed in the following way: (1) The diffusion calorisers must be tubular, and have a heating surface of at least double of that when direct steam is used. (2) The limed diffusion juices pass into the reheaters; heated by No. 2 steam, and issue from these reheaters at about 80° C. (176° F.), and are then carbon-

# CONCENTRATION OF BEET JUICE TO SYRUP 171

TABLE XXXII.—TABLE OF SECTIONS TO ADOPT FOR PIPES FOR THE PASSAGE OF STEAM FROM THE ESCAPE BALLOON TO No. 1, FROM No. 1 TO No. 2, FROM No. 2 TO No. 3, AND FROM No. 3 TO CONDENSER, FOR TRIPLE-EFFECTS OF DIFFERENT CAPACITIES. THE PRESSURE IN THE EXHAUST STEAM RESERVOIR IS 500 GR.,  $\frac{1}{2}$  KILO. PER SQ. CM. EFFECTIVE. THE VACUUM IS NIL IN No. 1, IT IS 32.4 CENTIMETRES IN No. 2, AND 58.13 CENTIMETRES IN No. 3 (CAMBIER).

A.	B.	C.	D.	E.
	Sq. Cm.	Sq. Cm.	Sq. Cm.	Sq. Cm.
1200	1.76	2.60	3.57	6.74
1600	2.31	3.36	4.70	8.81
2000	2.86	4.28	5.82	11.00
2400	3.52	5.20	7.14	13.58
3200	4.62	6.72	9.40	17.62
4000	5.72	8.46	11.64	22.00
4800	7.04	10.40	14.28	27.16
After this capacity speed of steam between No. 3 and condenser is brought to 80 metres per second.				
5600	8.14	11.92	16.54	27.16
6400	9.24	13.66	18.78	31.20

A, quantity evaporated in hectolitres in 24 hours in triple-effect. B, steam entrance section in sq. cm. into No. 1, speed of steam 30 metres per second. C, section of pipes in sq. cm. leading from No. 1 to No. 2, speed of steam 30 metres per second. D, section of pipes in sq. cm. leading from No. 2 to No. 3, speed of steam 30 metres per second. E, section of pipes in sq. cm. leading from No. 3 to condenser, steam speed 70 metres per second.

ated without further reheating. (3) The juices going to the second carbonation are heated to 100° C. in the reheaters, and go from thence to the second carbonation pans. (4) The juices going to the quadruple-effect are again brought to 100° C. (212° F.) in the reheaters by the steam from No. 1. (5) The syrups are likewise heated to 100° C. by No. 1, before filtration. (6) The vacuum strike pan is heated by steam coming half from No. 1 and half from No. 2, and its heating surface should be calculated accordingly. The whole system consists in giving to each vessel of the quadruple-effect the proportions suitable to the work it has to do, and passing all the liquids to be reheated into reheaters, independent of the pan into which they are run to be treated at the proper temperature. The diffusion calorimeters and the strike pan must have heating surfaces sufficient for the steam from the multiple-effect system to bring the liquids to the desired temperature, all of which involves much calculation, and an installation does not succeed well unless the proportions are exactly prescribed and observed. The reheaters used in this



installation are of two kinds: (1) *Austrian reheaters*.—In Austria the reheaters are rectangular and consist of vertical tubes packed between perforated plates, as in the vessels of the multiple-effect system, open from above to the air. The flow of the current of juice through them is rather slow, and the heating surface is also very great. (2) *French reheaters*.—In France closed reheaters are preferred, into which the pumps press so as to have a rapid current of juice. They may also be made of small dimensions, taking up but little room and working very regularly. Either system works well, the price of the plant alone differs; the installation is dearer in Austria than in France. But in each country each constructor has his predilections, and the engineer ought to humour his customers, so long as the final result does not suffer.

*Extraction and utilisation of the condensed water liquors from the individual vessels*.—The water condensed in No. 1 comes from the waste steam of the engines, which is used to heat that vessel, and from the direct steam from the engine boilers, which is added as an auxiliary if the engines do not furnish a sufficiency. This water is relatively pure, and is generally used to feed the boilers. The only impurity which it contains comes from the lubricants used for the steam cylinders. If *animal or vegetable* oils be used they are deposited on the tubes of the vessel with the condensed steam, and return to the boilers, where they may be dangerous on account of their corrosive action on the boilers. *Mineral* oils are therefore used, which are harmless. In No. 1 there is generally a pressure of  $\frac{1}{2}$  atmosphere. The condensed water runs naturally away into the feed-tank of the boilers.

*The ammoniacal vapour and ammoniacal pumps*.—The condensed waters from No. 2 and No. 3 vessels are charged with ammonia, because the boiling juice develops a great amount of ammoniacal vapour. These ammoniacal liquors are not much esteemed for feeding the boilers, because *the steam which they give off, being charged with ammonia, would corrode the copper taps, the safety valves, etc.* However, by using a portion of it only, these mishaps are not to be feared, owing to the distillation of the ammonia into a large quantity of steam. But a vacuum exists in No. 2 and No. 3, and to extract the water a pump is absolutely necessary; and, moreover, for No. 3 the pump should be well made, so as to cope with the high vacuum therein. These *ammoniacal pumps* should therefore be carefully constructed, because on their working depends the success of the system. Besides ammoniacal liquor there are also the gases coming from the juice, and the leakage air which enters through the joints. It is thus necessary to have—as Rillieux originally had—for each vessel an air-pump to cope with this double extraction.

*Rillieux's method of extracting the gases from the juice*.—Now the constructors of the first Rillieux plant, in making the first sets of

triple-effect plant, according to the plans brought from America, had neglected the removal of the air. The system therefore worked very badly, and it was by re-establishing the removal of the air, by a process other than the air-pump, that Rillieux was able, in 1882, to restore to the modern system the capacity of his old American plant. His system consisted in collecting the condensed water and the gases in a box in front of the ammoniacal pump, and in removing the air therefrom by a gauged tap, fitted with a pipe going into a space, where a greater vacuum existed than in the box, say in the following vessel. Only when this was done did the European plant work properly. They were then able to construct quadruple and quintuple-effects. Previous attempts failed owing to the mistake made in not removing the air which encumbered the vessels.

*Removal of ammoniacal gases.*—In addition to air and the heavy gases, which accumulate in the bottom of the intertubular spaces, above the water, which enables them to be removed into the receptacles into which these gases flow with the water underneath, there is also in the vessel a certain amount of ammoniacal gas, which is lighter than steam. These light gases accumulate in the top, under the upper tubular plate. But ammonia, in contact with copper, corrodes it, when a certain amount of air is present, as is the case in this instance. It has therefore to be removed as quickly as possible. That is done by making several apertures on the upper tubular plate, on which pipes are fixed, which coalesce into a single pipe, which emerges from the vessel on the outside (Fig. 46) and goes direct to the condenser. Owing to this arrangement the light gases are aspirated to the condenser, and thus attack the tubes to a less extent than if they were allowed to remain in contact with them. A gauge tap, already mentioned, between the armatures and the calandria, is used to regulate the flow of the ammoniacal gases. Witkowitz removes these ammoniacal gases (Fig. 56) in a special way.

*Mode of conveyance of steam from the engines and boilers to the multiple-effect.*—The triple-effect further includes the steam pipes which feed No. 1 with the exhaust steam from the engines, all of which passes into a cylinder, the escape-steam cylinder, to be led from there through a large pipe into the intertubular part of No. 1. The auxiliary direct steam from the boilers is led into the escape cylinder, or into No. 1 itself, by means of a balanced valve, which does not allow the steam to increase beyond measure the counter pressure in the escape cylinder, and thus stop the engines. The valve invented by Rillieux does not really work well, it is said, except when its tongue is itself balanced so as to allow a large escape of steam with a slight upward pressure. The valve constructed by Dulac answers well for this purpose.

No. 1 is best fed with juice by the pump.—The juice is pumped

into No. 1. There are other and more complicated feeding arrangements, but the pump best ensures regular feeding.

*No. 2 vessel aspirates the juice from No. 1, and so on.*—No. 2 aspirates the juice directly from the bottom of No. 1, and No. 3 from the bottom of No. 2, whilst the syrup also is extracted from the last vessel by a pump. Formerly this extraction was made by a *montejus* working exactly like the sampling vessel (p. 176). But the *montejus*, now almost everywhere condemned, deranges the work of the system at the moment of the vacuum.

*Mechanical filtration of the juice between No. 2 and No. 3 vessels to avoid encrustation.*—It is well to filter the syrup between the last and the second last vessel. To avoid extraction pumps, closed mechanical filters, e.g., Philippe's, are sometimes placed between the last two vessels in the path of the syrup intercalated on the feed-pipe. Sand filters, of which Stade of Berlin makes a speciality, are also intercalated and said to work successfully. This filtration has the effect of so far purifying the syrups as to greatly reduce the encrustations on the last vessel of the series.

*Cleaning the triple-effect apparatus.*—The different vessels of the multiple-effect system can be made to communicate with each other when it is necessary to empty them, which is only done when it is required to clean them or when the manufacture is finished. In America, Spenser used caustic soda to clean a "Yaryan," and found it to act effectively (see p. 263).

During the season 1899-1900 there were 255 sets of triple-effect plant in use in France, 93 sets of quadruple-effect, and 16 sets of quintuple-effect, whilst no less than 130 factories were using Rillieux's systematic method of reheating the juices by withdrawing the necessary steam from one or other of the vessels of the series forming the multiple-effect.

*Horizontal evaporation systems.*—All the foregoing refers to vertical, triple, and quadruple-effects. Rillieux's original plant (pp. 137 and 140) was horizontal, in the form of the tubular boilers of a locomotive. In Austria all multiple-effect plants are now constructed with horizontal vessels. The following are the points in which the horizontal differs from the vertical system.

*The drawbacks of the original Rillieux plant.*—Rillieux plant had several inconveniences. The height of the liquid was very great owing to the cylindrical form of the vessel, and the space reserved for the steam above the juice was too small, because the tubes rose higher than half the circumference. Again the tubes could not be dismantled, or only with difficulty, which was a great trouble in cleaning, and took more than one day on each occasion.

*How remedied.*—These defects were remedied by first making the vessel flat-bottomed, which gave it the appearance of a trunk or a Watt's boiler. The tubes, spread parallel with this flat bottom,

occupy but a small height for an equal heating surface. Owing to the small depth of juice the space for the steam was greater and the defects of the completely cylindrical vessel were avoided.

*The method of fixing the tubes in the flat-bottomed horizontal Austrian vessels.*—The manner of fixing the tubes is ingenious. The tubular plates in front and rear are pierced with holes of a wider diameter than the tubes. The latter pass through the plates, and on each is placed a vulcanised indiarubber washer. A cap taking eight tubes at a time presses with a single blow the eight washers against the tubular plates, and makes the joints tight. Eight tubes are therefore placed at a time. In cleaning, the cap, which is pressed and retained by a single bolt, is removed and the eight are at once freed. They are taken out to be cleaned, and a fresh set put in their place with their rubber washers and their cap. In a few hours the whole tubular portion of the plant may be renewed, thus saving time in cleaning. Only the rubber, not being fit to be used twice over, is so great an expense that these renewals are done as seldom as possible.

*The streaming process.—The Yaryan.*—The great obstacle to evaporation in all plant of this kind is the enormous mass of juice in circulation. Yet only the liquid which moistens the heated partition is evaporated; all the rest only retards the departure of the steam, which occasions perceptible delay in the process, and consequently diminishes the output of the evaporation plant. If there were only a thin layer of juice on the tubes, just enough to moisten the surface, the pans would evaporate much more. Attempts have been made to attain this object by causing the juice to stream over the surface of the tubes, leaving the interior empty for the escape of the steam. The first plant of this sort was invented by an American named *Lillie*, and caused but little commotion; whilst another American, *Yaryan*, adopting the same principle, made numerous claims in advertising his triple-effect, and obtained brilliant results in evaporating *water or pure liquors*. But the highly encrustating juices seem to have rendered his system impracticable. However, opinion is divided, the successes balancing the failures, so that it is difficult to get at the truth. See under "*Cane Sugar*" (p. 263), where the Yaryan gave good results at *Magnolia*, and where the encrustation was effectually treated with caustic soda; but the Yaryan at *Magnolia*, it is reported, has since been replaced by a *Rillieux*.

Attention being thus aroused, every one immediately tried to solve the problem by applying it to the existing plant. They placed on the tops of the vessels more or less well-conceived arrangements to distribute the juice in thin layers in the interior. But the matter is still in the experimental stage. However, it is possible the problem may be solved some day if encrustation does not form an insurmountable obstacle.

*Automatic sampling of the syrup.*—To ascertain exactly the nature of the virgin syrup to be boiled down to strike point, it has to be analysed. But the difficulty is in taking *regularly* a fair average sample of the syrup during boiling. To get exact results, Guerin devised the apparatus shown in Fig. 53. On the pipe leading from the filters to the strike pan store tank, a branch pipe A, 10 mm. in diameter, is fixed, to which is adjusted a tap B, connected with the valve C, wrought by a counterpoised lever arm to keep it usually closed. The valve exit terminates in a reservoir R, of 1 to 2 litres capacity, ending in its lower part in a stop-cock H. The six-toothed wheel D lifts the arm of the valve F, every time it is set running, by the indices G and G', fixed on another wheel E. The index G' is shown as not working on the illustration. The indices are fixed on the sides of the toothed wheel E. The wheel E is driven by an escarpment, the motion of

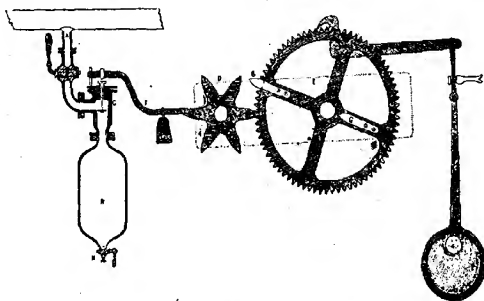


FIG. 53.—Guerin's automatic syrup sampler.

which is due to an eccentric, as shown in the drawing. This escarpment may also be driven by a belt arrangement or by clock-work. *Manner in which the apparatus works.*—The tap H of the reservoir is closed, and the tap A opened. The large wheel E is driven at such a speed as to make one revolution every three minutes. If the index G be alone arranged for working, the small wheel D will make one revolution every 18 minutes; but as it only carries one stud which can work the arm of the lever F and the valve C, the valve only opens and shuts every 18 minutes. If the average sample obtained in this way be not enough its volume may easily be doubled by placing the two indices G in the same position, the wheel D will then make a complete revolution every nine minutes, and the valve will lift itself twice as often as when only one index is at work. This apparatus eliminates all errors in taking the sample whether they be accidental or intentional. Another syrup sampling device is described on p. 158.

## CHAPTER V.

### BOILING DOWN OF THE BEET SYRUP TO CRYSTALLISING POINT IN VACUUM PANS — CRYSTALLISATION IN MOTION — THE TREATMENT OF WEAK LIQUORS AND OF LIQUORS OF BAD QUALITY.

THE following tables of the solubility of sugar solutions at different temperatures, as well as the boiling-point of sugar solutions at different pressures of the air, will be useful for reference, and well merit careful study at the very outset. Although they speak for themselves very plainly and require little or no explanation, yet perhaps the following remarks may serve to put the reader on his guard against being misled by failure to read these and similar tables that occur throughout this and other treatises aright. In the first place the density of a solution of sugar or of any other liquid is of no value whatever unless the temperature at which the determination is made be given. Even those *who ought to know better* confuse densities at say 180° F. with the density of the same solutions at 60° F., whereas the least exercise of common sense would tell those who fall into such a pit that a gallon of well on to boiling sugar liquor weighs less than a gallon of sugar liquor at say the freezing-point. Again, boiling-points are of no value unless the barometric pressure in inches (or other unit) be given. But it would moreover be quite unreasonable to expect either concentrated beet juice or syrup from the triple-effect or raw sugars to show the same density per unit per cent. of sucrose as solutions of pure crystallised sucrose itself. Still more irrational would it be to expect to be able to calculate to the fourth decimal place, *i.e.*, to one part in 10,000, the percentage of sucrose in a raw sugar liquor from mere density alone, irrespective of temperature and the nature of the sugar in the first instance. Yet instances are not wanting again amongst those who ought to know better where this has been done. Tables therefore are only of value when rationally used to make rational deductions. Much confusion arises in the sugar industry from the use of Baumé's hydrometer, of which there are so many different graduation scales extant, which differ from each other to such an enormous extent that the German Baumé actually shows 4 per cent. more glycerine in the same sample than the French Baumé! Baumé's erratic scale.

should give way to Twaddell's. But the literature of sugar abounds in densities according to Baumé, and in the confusion of scales prevalent, degrees Baumé, although initially readily intelligible and serviceable enough in a comparative way to those using them, are only a very rough guide indeed to those who are unaware of this extreme divergency in the different Baumé's scales, the misleading nature of which is aggravated by those who quote such scales invariably neglecting to mention which particular scale they have adopted.

TABLE XXXIII.—SOLUBILITY OF SUCROSE AT DIFFERENT TEMPERATURES, WITH DENSITIES IN DEGREES BAUMÉ AND DEGREES TWADDELL, AND IN ACTUAL GRAVITY AT THE TEMPERATURE OF THE EXPERIMENT, AND AS BROUGHT TO 15° C. (FLOURENS).

Temperature °C.	Sugar per Cent.	Degrees Baumé.		Degrees Twaddell.		Actual Gravity.	
		At the Temperature of the Experiment.	At 15° C.	At the Temperature of the Experiment.	At 15° C.	At the Temperature of the Experiment.	At 15° C.
0	64.70	35.30	34.60	64.70	63.00	1.3235	1.3150
5	65.00	35.35	34.90	64.86	63.80	1.3243	1.3190
10	65.50	35.45	35.20	65.10	64.50	1.3255	1.3225
15	66.00	35.50	35.50	65.20	65.20	1.3260	1.3260
20	66.50	35.60	35.75	65.50	65.80	1.3275	1.3290
25	67.20	35.80	36.25	66.00	67.11	1.3300	1.3355
30	68.00	36.00	36.70	66.50	68.10	1.3325	1.3405
35	68.80	36.20	37.10	67.00	69.20	1.3350	1.3460
40	69.75	36.40	37.50	67.50	70.20	1.3375	1.3510
45	70.80	36.75	38.10	68.20	71.80	1.3410	1.3590
50	71.80	37.10	38.70	69.20	73.20	1.3460	1.3660
55	72.80	37.50	39.30	70.20	74.80	1.3510	1.3740
60	74.00	37.90	39.90	71.20	76.40	1.3560	1.3820
65	75.00	38.30	40.55	72.30	78.20	1.3615	1.3910
70	76.10	38.60	41.10	73.00	79.60	1.3650	1.3980
75	77.20	39.00	41.70	74.00	81.20	1.3700	1.4060
80	78.35	39.30	42.20	74.80	82.60	1.3740	1.4130
85	79.50	39.65	42.60	75.80	84.40	1.3790	1.4220
90	80.60	39.95	43.30	76.40	85.80	1.3820	1.4290
95	81.60	40.10	43.70	77.00	86.80	1.3850	1.4340
100	82.59	40.30	44.10	77.50	88.00	1.3875	1.4400

*Preliminary filtration.*—The syrup as it comes from the triple-effect evaporation plant has a density of 25° to 30° Baumé, sp. gr. 1.2 to 1.25, that is to say, during that operation 80 to 85 per cent. of the water which it contains is removed as steam. It is now necessary to concentrate it to the crystallisation-point. At the outset it is filtered, because it is turbid. The methods of filtering the

syrup are identical with those for the juice, whether through animal charcoal (Fig. 41) or mechanical filters (Figs. 44, 45A, 45B). Filtration through animal charcoal is, however, almost totally abandoned. The filtered syrup is collected in a store tank, from which it is drawn by the sugar boiler. The operation of boiling consists in evaporating and concentrating the syrup to crystallisation-point in an apparatus called a vacuum pan. It is a pan provided with a steam coil, in which the evaporation process is finished in the vacuum produced by a condenser and an air-pump, as in the case of the triple-effect.

*Rillieux economises steam in heating the vacuum pan.*—It has been mentioned that in Rillieux's new process boiling was effected by using the steam of the Nos. 1 and 2 of the triple-effect to evaporate the mass to strike-point. In order to work in this way a large heating surface is required, and vertical strike pans are not at all adapted for that kind of work.

1. *Horizontal pans.*—The kind of pan suitable for utilising Nos. 1 and 2 steam.—The pans used for this purpose are horizontal, of the same construction as the vertical, only the flat bottom does not answer well for evacuation purposes.

*The Lexa types of horizontal vacuum pans.*—Austrian models.—Lexa, who made a special study of the construction of horizontal strike pans, has made two types of vacuum pans in Austria. 1. In his first model the flat bottom is replaced by three cones, each of which is provided with a steam coil to heat the mass in the cone, and a discharge door. 2. In the second much more elegant and practical type, the flat bottom remains, but it consists of two movable doors, which slide horizontally under the pan, to the right and to the left, so as to leave the whole of the bottom wide open, and thus let the whole mass flow out rapidly. These horizontal pans, monumental in style, gradually replacing all the vertical pans formerly in existence in Austria, are slowly being adopted in France. They constitute a real step in advance, both from the construction point of view and that of a more economical utilisation of the factory steam, and also because the boiled mass has a better appearance and the yield is higher.

*Less injury from steam heat in horizontal vacuum pans than in vertical.*—In fact, with direct steam in the coils of vertical pans (Fig. 54) the syrup is burned, the steam is too hot, therefore the masses cuites are very dark in colour. With horizontal boiling, on the contrary, the steam is cooler, does not darken the syrup at all, and the masse cuite is white and rich.

*Rillieux's original vacuum pan a horizontal one.*—Moreover, the first Rillieux vacuum pans were horizontal and heated by No. 1 steam, and the reports of 1845 relate that the masses cuites were crystalline and blonde—a result which is unattainable with Howard's vacuum pan.



TABLE XXXIV.—SOLUBILITY OF SUCROSE IN WATER AT DIFFERENT TEMPERATURES—(HERZFELD).

°C.	A.	B.	°C.	A.	B.	°C.	A.	B.
0	64.18	179.2	34	69.38	226.6	68	75.80	313.5
1	64.31	180.3	35	69.55	228.4	69	76.01	317.0
2	64.45	181.4	36	69.72	230.3	70	76.22	320.5
3	64.59	182.5	37	69.89	232.3	71	76.43	324.4
4	64.73	183.6	38	70.06	234.2	72	76.64	328.3
5	64.87	184.7	39	70.24	236.1	73	76.85	332.2
6	65.01	185.8	40	70.42	238.1	74	77.06	336.0
7	65.15	187.0	41	70.60	240.2	75	77.27	339.9
8	65.29	188.2	42	70.78	242.3	76	77.48	344.4
9	65.43	189.3	43	70.96	244.4	77	77.70	348.8
10	65.58	190.5	44	71.14	246.6	78	77.92	353.2
11	65.73	191.8	45	71.32	248.7	79	78.14	357.6
12	65.88	193.1	46	71.50	251.0	80	78.36	362.1
13	66.03	194.4	47	71.68	253.3	81	78.58	367.1
14	66.18	195.7	48	71.87	255.7	82	78.80	372.0
15	66.33	197.0	49	72.06	258.0	83	79.02	376.9
16	66.48	198.4	50	72.25	260.4	84	79.24	381.9
17	66.63	199.7	51	72.44	262.9	85	79.46	386.8
18	66.78	201.1	52	72.63	265.5	86	79.69	392.6
19	66.93	202.5	53	72.82	268.0	87	79.92	398.4
20	67.09	203.9	54	73.01	270.6	88	80.15	404.2
21	67.25	205.4	55	73.20	273.1	89	80.38	409.9
22	67.41	206.9	56	73.39	276.0	90	80.61	415.7
23	67.57	208.4	57	73.58	278.8	91	80.84	422.3
24	67.73	209.9	58	73.78	281.6	92	81.07	428.8
25	67.89	211.4	59	73.98	284.5	93	81.30	435.4
26	68.05	213.0	60	74.18	287.3	94	81.53	442.0
27	68.21	214.7	61	74.38	290.4	95	81.77	448.0
28	68.37	216.3	62	74.58	293.5	96	82.01	456.3
29	68.53	217.9	63	74.78	296.7	97	82.25	464.0
30	68.70	219.5	64	74.98	299.8	98	82.49	471.7
31	68.87	221.3	65	75.18	302.9	99	82.73	479.4
32	69.04	223.1	66	75.38	306.4	100	82.97	487.2
33	69.21	224.8	67	75.59	310.0			

A, sugar per cent. in resulting solution. B, 100 parts of water dissolve of sugar to give A.

TABLE XXXV.—BOILING-POINTS OF SUGAR SOLUTIONS IN VACUUM PAN AT DIFFERENT PRESSURES OF AIR.

Bar in.	°F.	°C.	°R.	Bar in.	°F.	°C.	°R.
0.74	115	46.11	36.89	2.05	150	65.55	52.44
0.86	120	48.89	39.11	2.36	155	68.33	54.67
1.01	125	51.67	41.35	2.72	160	71.11	56.88
1.17	130	54.44	43.56	3.10	165	73.89	59.11
1.36	135	57.22	45.78	3.52	170	76.67	61.33
1.57	140	60.00	48.00	4.00	175	79.44	63.56
1.80	145	62.78	50.22				

## BOILING DOWN BEET SYRUP TO STRIKE POINT 181

2. *Vertical strike pans.*—The vertical vessels (Fig. 54) are cylindrical, surmounted by a dome truncated on a plane perpendicular with the axis, in the centre of which is a door of 20 to 30 inches through which the *masse cuite* is discharged. This door is closed by contact against its armature, both door and armature being perfectly smooth, and sliding the one on the other horizontally round an

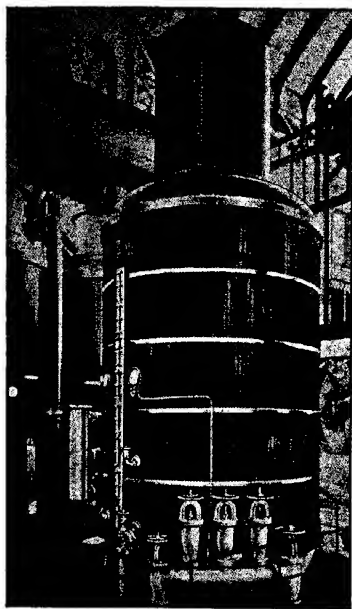


FIG. 54.—Vacuum strike pan (GAIL).

eccentric axis in opening for discharging. There are generally three or four steam coils in the pan, touching the sides, as far as the door, and an additional coil in the centre. Each steam coil has its tap on the outside, but all branch off from one main pipe receiving its steam direct from the boilers. Fig. 55 shows how the calandria is fitted with a steam lyre in place of a coil.

*Wilkowicz's radiating heater.*—Impressed with the great amount

of heating surface left idle in all evaporating vessels heated by coils or lyres, and with the great benefits which would accrue from circulation of the juice, Witkowicz, after trying mechanical motion and injection under pressure, designed his radiating heater, which lends itself to numerous applications in reheating and carbonatation tanks, and in evaporation and vacuum pans. It consists of a steel-forged body in one piece, and completely closed. Two series of X tubes

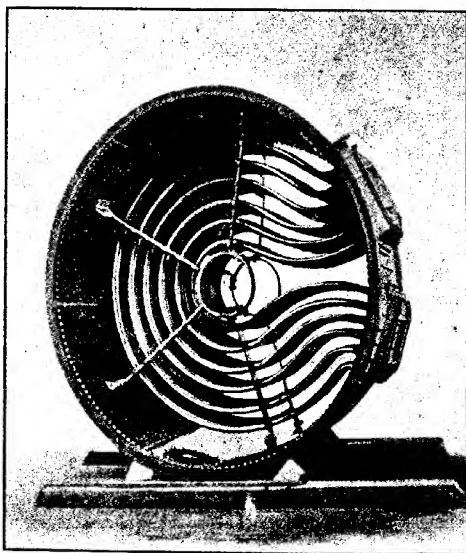


FIG. 55.—Calendria of strike pan, being fitted with steam lyres in the Cail workshops.

(Fig. 55) cross the body and impart to the juice, which passes over them, highly divided projections in an inverse direction, which produce an absolutely uniform circulation in the vessels where the radiating heater is fixed. The ammoniacal vapours and the condensed water are easily and surely removed. It may be applied to existing evaporation vessels, or to tubular vacuum pans, so as to increase the production of the apparatus by more than 30 per cent.

*The radiating heater applied to the "firsts" vacuum pan.—All the*

old types of "firsts" vacuum pans had insufficient heating surface, but with the radiating heater it is claimed that the heating surface may be doubled or even trebled whilst still retaining one or two coils in the bottom of the vessel to form the grain. Fig. 56 shows two radiating heaters, installed in an ordinary vacuum pan, replacing a removed coil. Radiating heaters may be heated by high pressure steam or juice steam, and their adoption gives great elasticity to the work. The circulation of the *masse cûle* becomes very energetic,

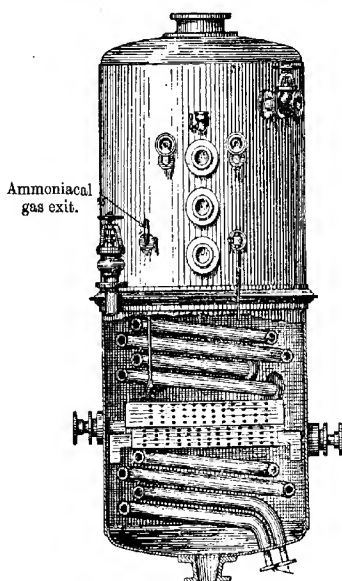


FIG. 56.—Radiating heater applied to a vacuum pan replacing a removed coil (MAGUIN). See Fig. 36 (B).

by the agitation which is imparted to it in two different directions in the tubes of the radiating heater. This motion facilitates crystallisation. Exhaustion is better, and the time taken in boiling is very much reduced.

*The radiating heater applied to the "seconds" vacuum pans.*—In working the "seconds" the vacuum pans have a still smaller heating surface than those intended for "firsts," and the radiating heater brings a great improvement to bear on their interior arrangement

and in the working of the product. Numerous improvements have been made in Germany and in Russia on existing "seconds" vacuum pans, generally tubular, with small coils and of horizontal shape. A "seconds" jet vacuum pan with motion may be fitted with the radiating heater so as to realise all the advantages of Hach and Laucke's mixers without their numerous drawbacks, the most serious of which is that when it is desired to continue the boiling in these vessels there is insufficient and badly arranged heating surface.

The cylindrical portion of the vertical vacuum strike pan carries oblong lenses, arranged on a frame running up the whole height of the pan (Fig. 54). It has, like the pans of the triple-effect, its *butter* tap, a second larger tap for breaking the vacuum by allowing the air to enter, so that the mass may be evacuated, and a pressure gauge indicating the vacuum in the interior. Finally, on the lower side, well within reach of the boiler, is a proof-stick to take a sample of the mass, and to judge of the state of the boiling. The proof-stick is simply a long rod, 2 to 2½ inches in diameter, dipping into the mass, and sliding by gentle friction in a bronze armature or sheath. On a point of the rod near its extremity is a depression. When the proof-stick is drawn towards the operator the depression comes *outside* the armature or sheath, the end of the proof-stick still corking up the opening. In the depression there is left a sample of the boiled mass, and from this sample, which is removed by the finger, the condition of the mass being boiled is ascertained.

*Boiling to proof.—String proof.*—Formerly boiling was done in open pans over a naked fire. The progress of the operation was judged by the appearance, between the finger and thumb, of a drop of liquid taken as a proof. If this drop, pressed between the index finger and thumb and the two rapidly drawn apart, formed a long, continuous, unbroken thread, it was said to have got to the stringing-point. Hence the term, boiling to string proof; if the thread broke, forming a longer or shorter hook, it was said to have got to the *light hook* or the *strong hook*. The latter was the sign of the finish of the boiling; the syrup, then only containing 12 to 15 per cent. of water, was run into tanks or moulds, which were left in a stove, and at the end of a certain time the whole crystallised. With closed boiling pans, working *in vacuo*, the boiling is done by the grain.

*Feeding the crystals.*—When several successive additions of lighter syrup are added to the boiling mass, small, almost imperceptible crystals are formed in it, and if the feeding with this syrup be continued the crystals grow, until they become as large as a crystal of coarse salt. By feeding in this way the grain is nourished without forming any additional crystals, and the skill of the boiler is to know how to obtain regular-shaped crystals, all of the same size.

This is called *boiling in grain*. When the pan is quite full of this mass of grains, moistened by a scanty liquid called molasses, the *masse cuite* (boiled mass) has been obtained, and the process is pushed as far as possible by evaporating the last portions of water from the molasses, so that the *masse cuite* only contains 5 to 6 per cent. of water.

*Discharging the contents of the vacuum pan into shallow tanks.*—When this occurs the operation is finished. The engine is stopped, the vacuum broken, the discharge door opened, and the mass falls into flat tanks, underneath, arranged so that they may be easily emptied.

*Boiling in grain.*—Such is summarily the method of boiling in grain, which yields a crystalline mass directly, and which considerably abridges the time occupied by the old methods in open pans, whilst it, at the same time, yields better and whiter products. During the season 1899-1900 there were 540 vacuum pans of enormous capacity in use in France for boiling in grain (*à cuire en graine dans le vide*).

*"Simplex" method of feeding "seconds" with "firsts" crystals, and subsequent treatment of masse cuite thus obtained by crystallisation in motion.*—The problem to be solved was: It being given that with the return of the drainage liquors to the "firsts" vacuum pan and with crystallisation in motion there is obtained on turbinating a poor drainage liquor of 66° to 68° purity, what is the most simple and the most rapid method of obtaining in one operation the whole of the crystallisable sugar and the molasses of 55° to 58° purity? Maguin claims to have obtained the desired result by means of (1) A previous feeding of the "seconds" *masse cuite* by means of the "firsts" *masse cuite*. (2) Methodical cooling, regular throughout the mass, obtained in Ragot and Tourneur's exchange of temperature mixers. Fig. 57 shows the arrangement adopted for feeding. As soon as the boiling of the "firsts" is finished and all the drainings have been returned, the vacuum is broken in the "firsts" vacuum pan; the valve E is opened, and the vacuum being maintained in the small apparatus, the number of hectolitres required for feeding is aspirated into the latter. A gauge glass shows the number of hectolitres run in. When the feeding is finished the valve E is closed, the vacuum is started again, and the boiling of the "firsts" goes on as usual. The drainage liquor to be boiled is then introduced into the "seconds" apparatus slowly. It is thus exhausted of its sugar during the whole duration of the boiling by the feeding crystals previously introduced. This operation, when well conducted, produces a rather complete exhaustion of the drainage liquor, which has now only to be aided by cooling the sugar, dissolved in the *masse cuite* in consequence of the high temperature. In fact, the mother-liquor used for feeding, drawn from the "firsts"

*vacuum pan*, is appreciably of the same purity as the drainage liquor to be boiled, which comes from itself, and is nothing other than the mother-liquor of a preceding boil. As soon as the boiling of the "seconds" is started, the exhaustion, which goes to increase the regular and well-divided feeding crystals, in the mass commences immediately, and is carried on during the whole duration of the boiling. If, therefore, the drainage liquor to be boiled has  $68^{\circ}$  of

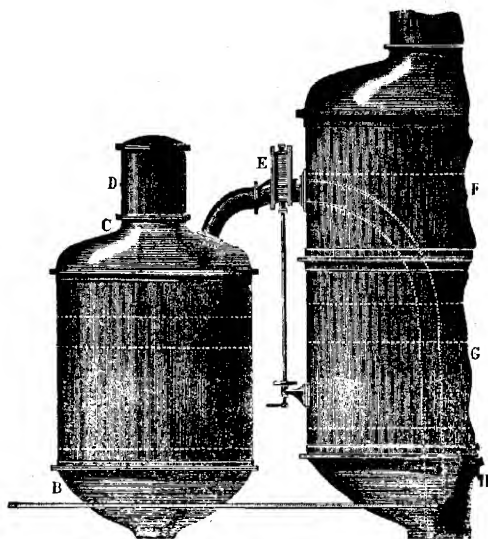


FIG. 57.—Method of feeding the "seconds" *masse cuite* with crystals from the "firsts" (MAGUIN).

purity, it, can easily be reduced to  $63^{\circ}$  to  $65^{\circ}$ , according to its viscosity. At this moment, the boiling being finished, the mass is cooled in a "crystallisation in motion cooler," and here a digression must be made from the description of cooler used in this special case to make a few historical remarks in regard to crystallisation in motion and the principle on which it depends. The first machine in which this principle was put into practice was the Bocquin-Lepchinsky cooler.

*Crystallisation in motion mixing coolers.*—A system, only a few years old, and which is now much used, is the crystallisation in

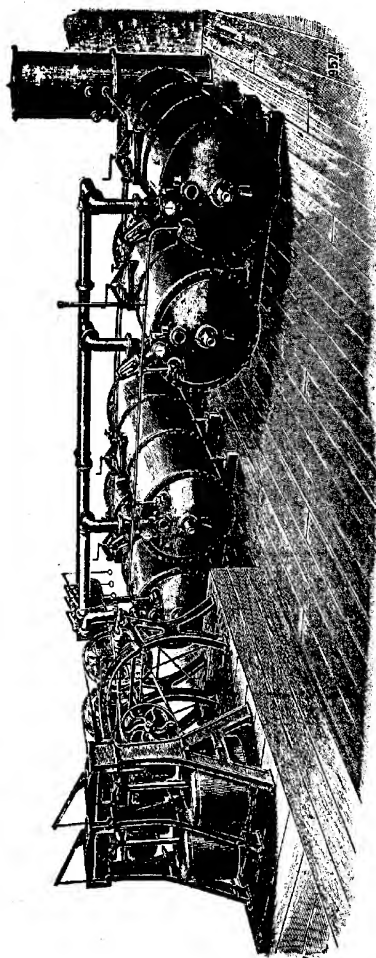


FIG. 53.—General arrangement of cane-sugar curing plant. Crystallisation in motion process—(Watson, Laidlaw & Co.).



motion cooler. It is a large tank with a round bottom, in the horizontal axis of which an agitator revolves. A current of cold water circulates in an outer jacket. This tank has the capacity

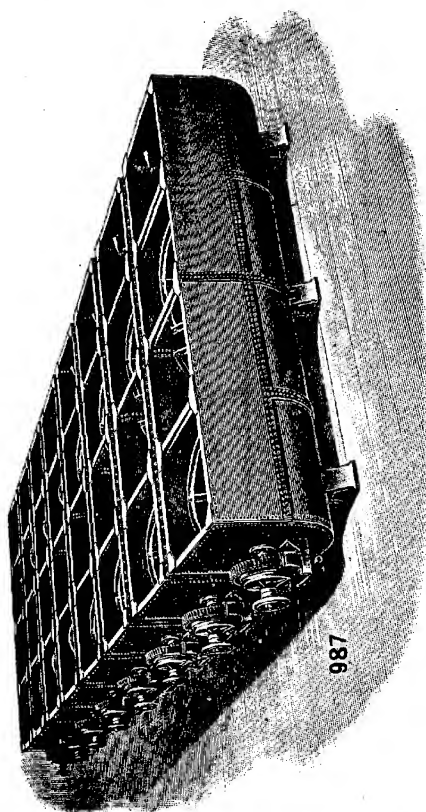


FIG. 63.—Eight steel plate crystallisers (open type)—(WATSON, LAIDLAW & CO.).

of a vacuum strike pan, and, during the whole of the time occupied in cooling, the agitator is kept in motion. The result is that, in spite of the cooling, the crystals remain detached from each other, and

are still fed; whilst in ordinary tanks, or sugar boxes, the *masse cuite* solidifies, thus preventing regular crystallisation. The method thanks to this motion, gives therefore a greater yield and larger crystals than cooling in tanks.

This method is called crystallisation in motion, and its principle is applied to all the products of the sugar factory, whilst originally it was only applied to the "firsts" *masse cuite*.

*The principle on which the phenomenon depends.*—If crystallisable molasses, to which a piece of sugar crystal has been added, be stirred, the crystal increases, and a result is obtained after a few hours' mechanical working which could only be got after the expiry of several months by the old process. This is a step in advance which is occupying attention, but which is not known generally enough to enter completely into the domain of actual practice. It

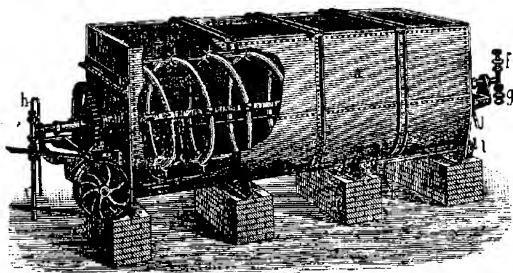


FIG. 60.—Crystallisation in motion cooler with revolving coil, etc., in which current of cold water circulates—(RAGOT AND TOURNEUR).

is only in the experimental stage, but these experiments have been so successful that there is every reason to hope for a great future for crystallisation in motion. In 1899-1900 there were, in fact, 191 factories in France working with *open crystallisation in motion* (Fig. 59), and 20 employing the same principles in *closed vessels* (Fig. 58).

*Crystallisation in motion cooling mixers* (Fig. 60).—But to return to the "seconds" *masse cuite* as discharged from B (Fig. 57). It is run into a variation of a crystallisation in motion cooler, in this case the Ragot and Tourneur. Contrary to all other systems, in which cooling is done by a surface exposed to contact of the surrounding air or the circulation of cold water on the outside by a cold-water jacket, in the Ragot and Tourneur system the cooling is done by exchange of temperatures between, on the one hand, a revolving coil in the centre of the mass, and in which a current of water circulates,

and, on the other hand, the *masse cuite* itself put in motion by blades fixed on the coils. There is thus a regular and active exchange of temperature, which induces an active and uniform lowering throughout every point of the mass, and which may be made as slow and as rapid as desired by opening or closing more or less the discharge taps of the water flowing in the coil. By treatment, in the Ragot and Tournour apparatus, the temperature of the "seconds" *masse cuite* may be lowered from 80° to 85° C. to 30° to 35° C. in twenty-four to thirty hours. The exhaustion thus obtained brings the mother-liquor from 63° to 65° to 56° to 58° purity, that is to say, molasses. The *masses cuites* thus treated are easily turbinéd and yield brown sugar, saleable at 90 to 92 per cent. Where, for particular reasons, it is not desired to run the drainage back to the "firsts" vacuum pan and to produce extra sugars, the simplex system is applied to the "thirds," that is to say, that the latter is fed with the crystals of the "seconds". In that case the "seconds" and "thirds" are mixed in the cooling mixers, and a less total volume is required. The result obtained is always the same. Ragot and Tournour's mixers represent for equal volume, from the point of view of rapid cooling, three jacketed mixers and five to six simple mixers, *i.e.*, it requires three to six times less to obtain the same result. In the working of the "firsts" the cooling from 85° to 40° C. is done in six to eight hours.

*The older method of cooling at rest in larger shallow tanks and in sugar boxes.*—The boiled mass, according to what has been said, is simply syrup, of which a large portion has separated out in the form of crystals. The molasses which moisten these crystals contain, besides sugar, all the impurities of the defecated juice. If the boiled mass be allowed to cool, the sugar which it contains in solution still continues to crystallise round the existing crystals by molecular attraction. But the crystals are not so pure and are less brilliant than those contained in the hot mass. Nevertheless, it is advantageous, from the point of view of immediate production, to allow the mass to cool, if regard be not had to an extraordinary beauty in the crystals. Certain plans have been made to effect this cooling under the best possible conditions. The simplest way is to place large shallow tanks underneath the vacuum pan, and to let the boiling mass drop into them.

*The utility of the sugar boxes and tip-tilting trucks.*—Another method often used is cooling in sugar boxes (Figs. 61-63). The mass is run into a series of small truncated boxes, which are charged with only 2 cwts. These are stored in a cool room until completely cold. This involves much handling, but they present great advantages, by facilitating the turbine process, as will be described in the sequel. They are handled by means of small trucks, consisting of two large wheels, between which each tank can be caught by lugs fixed on

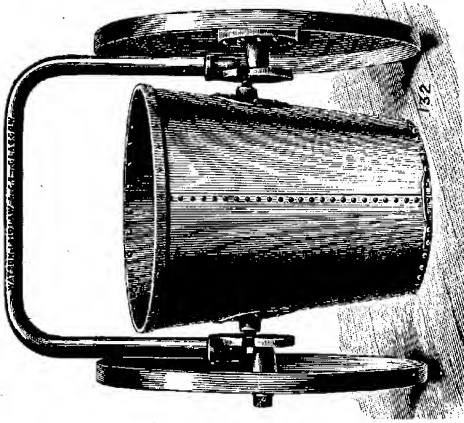


FIG. 61.—Method of raising sugar box for transport. The unique method of discharging these boxes is shown in Fig. 70. Other styles, also of Watson, Laidlaw & Co.'s construction, and selected from several varieties constructed on different principles, are shown in Figs. 63 and 67-9.

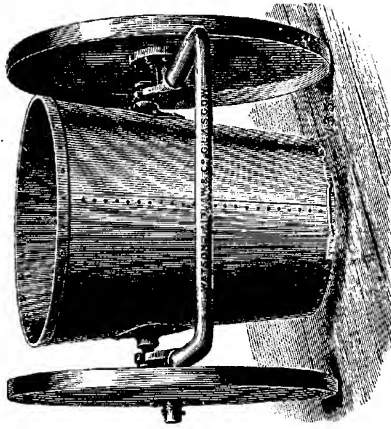


FIG. 62.—Sugar box raised into position for transport. Other styles, also of Watson, Laidlaw & Co.'s construction, are shown in Figs. 63 and 67-9.

it for the purpose. They are thus easily transported, and can be pivoted on themselves by completely tilting them, when it is desired to empty them, without assistance and without any great exertion.

*Difficulties in boiling, and juices impossible to boil.*—These are due to (1) presence of gummy substances in the juice; (2) imperfect liming; (3) fermentation of the juice; (4) excessive alkalinity of the syrups. In (1) and (2) boiling is slow and difficult, but the grain can always be formed. In (3) and (4) it is impossible to do so without a deal of manœuvring. (1) The first is caused either by the beets

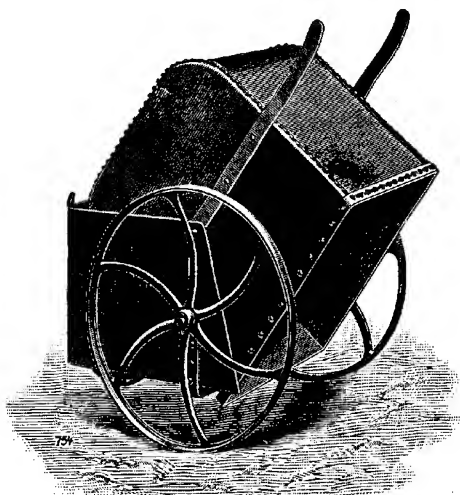


FIG. 68.—Square tipping truck for *masse cuite*, which can be used with a "concrete sugar" plant (WATSON, LAIDLAW & Co.).

being spoilt by too long storage in silo, a common mishap at the end of the season, or to excessive heat in certain countries south of France, Spain, etc., in the beginning of the season, rendering it altogether impossible to preserve the beets. The ferments generated in the beets partly transform the sugar into glucose, and also generate exceedingly viscous products. Soon after boiling has begun the vacuum rises to 20 to 21 inches; the syrup scorch and the air bells have great difficulty in escaping. To form the grain it is necessary to withdraw almost all the injection water so as to lower the vacuum as far as possible and to feed by small and frequent gushes. The

vacuum gradually lowers, small crystals appear, on the glass, surrounded by a gummy liquid, which holds the crystals like the meshes of a net and render the nutrition of the grain very difficult. Boiling must be slow and regular. The syrup darkens to a black brown. As to pressing the *masse cuite*, it can only be done very badly and always incompletely. The *masse cuite* must be turbinated rapidly, for if it begins to cool the sugars purge with difficulty, and it is then necessary to further refine, which means a further loss of white sugar, to avoid which, in hot countries, beets must not be taken delivery of in advance, or a judicious mixture must be made of sound and spoiled beets so as to diminish the bad effects. (2) A difficulty in boiling, owing to a *deficiency of lime* in the first carbonatation, is immediately manifested. The syrup tanks are covered with an abundant white scum. In the strike pan the syrups froth enormously, with a tendency to prime. A jet of direct steam can alone be used under such conditions; the evaporation is therefore very slow. The froth may be partly suppressed, either by introducing grease, oil or melted tallow into the vessel, or a little dilute hydrochloric acid, which instantly suppresses the froth. It is advisable to add it to the syrup tanks. As soon as the froth has been suppressed the steam may be increased, and evaporation is then effected more rapidly; the grain is easily produced, and the boiling process is pursued as under normal conditions. One must expect abundant frothing in the boiling of low-quality products, and also on the filling vessels, which is easily suppressed, as will be seen farther on. (3) The third case is due to the fermentation of the juice after a temporary stoppage of the factory (breakdown of the machinery, muddy diffusion water, etc.), or the starting of a pulping machine. In the strike pan the steam does not take hold upon these fermented juices, the manometer rises to 4 or 5 atmospheres, the vacuum rises to 25 or 26 inches, the juice remains cold, and a thick scum rests on the surface of the liquid. To remedy this the only practical method to use is to pour into the strike pan 1 litre of a 5 per cent. solution of carbolic acid. The effect produced is immediate. The scum disappears, the vacuum falls down again to 17 to 18 inches, and the manometer makes a sudden backward movement. The liquid enters into ebullition, and the boiling, which was immobile and impossible, is as easily conducted as in an ordinary boiling. It is well to add a little carbolic acid to the syrup tanks, so as to anticipate the work. (4) If the difficulty be due to an excess of lime, the analysis of the syrup will at once detect it. The steam, as before, does not take hold on the juice, the manometer remains at 4 or 5 atmospheres, the vacuum at 26 to 27 inches, the syrup remains cold, but no surface froth is produced. Hydrochloric acid, the commercial acid used for washing the triple-effect, must then be used to saturate the excess of lime. The operation is commenced by running 1 to 2 litres of the acid into the

boiling vessel, stopping when the manometer is seen to redescend. Ebullition soon takes place. If it be seen that the effect is satisfactory it is useless to add more acid, as the result would be to wash the vessel, the juice would become black and be contaminated with particles of iron or scoriæ, which would again contaminate the *masse* and the sugar. It would be advisable to add some to the syrup tanks. This would greatly facilitate working. In spite of these precautions boiling is slow, and concretion difficult—lasting two hours, and very imperfect. It is thus much preferable to make small boilings so as to gain time, because a boiling to the full capacity of the apparatus would require 17 to 18 hours.

*Difficulty in boiling low-quality products.*—To remedy this, add to each boiling 1 litre of carbolic acid in 1 per cent. solution.

*Analysis of syrups and masses cuites.*—*Sampling syrups.*—The fair average sample of *syrup* obtained by Guerin's sampler (Fig. 53) is well triturated to ensure thorough admixture.

*Precautions in sampling the masses cuites.*—The sample must be taken from the actual flow of the *masse cuite*, because when it is deposited in the tanks in the fill-house it is impossible to take a fair sample, as the crystals commence to deposit immediately on the bottom of the vats. Some *masses cuites* deposit very fine crystals; it is therefore advisable to triturate the sample taken, so as to get a uniform mass. The "seconds" *masse cuite* often produces, on stirring, an abundant granular crystallisation, and the whole mass assumes a crystalline appearance, like that of honey. These small matters require careful attention, else a fair average sample for analysis will not be obtained.

*Analysis.*—Either 50 c.c. or 50 grammes are taken for analysis, according as the result is to be expressed per litre or per 100 kilogrammes. Fifty grammes of *masse cuite* are invariably taken, dissolved in distilled water, and after cooling made up to a volume of 250 c.c. The liquid thus obtained, being a 20 per cent. solution, may be used for all the estimations that have to be made. The density of the liquid at 15° C. shows the apparent percentage of dissolved matter just as well as the density of the *masse cuite* itself.

1. *Sugar estimation.*—A solution of basic acetate of lead is added to 100 c.c. of the solution prepared as above so as to make up the bulk to 110 c.c. Instead of multiplying the degrees read off by the normal weights, they are multiplied by the following coefficients: 0.896 for the French saccharimeter and 1.433 for the German one.

2. *Ash.*—To avoid calculation, the works chemist should construct tables accordingly. From 5 to 10 c.c., corresponding to 1 or 2 grammes of substance respectively are taken, 2 drops of sulphuric acid added, the whole evaporated on the water bath, and the dry black mass is incinerated in a muffle furnace.

3. *Purity and saline coefficient.*—These are calculated from the

analytical data obtained. To get the real, actual purity an exact moisture determination must be made, working on 10 c.c. of liquid. This determination is made by Josse's striated filter paper method, rolled on a bobbin having the same dimensions as the capsule. The paper absorbs the liquid, and spreads it over a considerable surface. It is dried at 105° C. until of constant weight.

4. *Dissolved matter and non-saccharine.*—The actual dissolved matter (Table XXXVII.) may be approximately determined from the apparent percentage of dissolved substances thus: The amount of non-saccharine is determined, which is multiplied by the coefficient 0.93 and added to the sugar. The result is very near the actual amount of substances dissolved. By subtracting it from 100 the remainder gives the water in the sample tested.

5. *Alkalinity.*—The alkalinity is determined on 50 c.c., using sensitive litmus paper as indicator.

6. *Total lime.*—The total lime is determined by means of the standard soap (hardness) test on 20 c.c. diluted with its own bulk of distilled water. The degrees are multiplied by 0.0057 so as to get the percentage of CaO, the circumstances being quite identical with the analysis of a water, the proportion 20 c.c. = 4 grammes per 100 = 40 per 1,000. Sidersky's burette (p. 99) is so graduated as to render any calculation unnecessary.

7. *Determination of the quantity of crystals in a "firsts" masse cuite.*—The remainder of the sample taken for analysis is placed in a funnel, the stem of which has been loosely packed with glass wool or asbestos fibres. A previously tared platinum capsule is placed underneath, to collect the raw syrup adhering to the crystals. The capsule is weighed in order to get the weight of syrup; a drop of sulphuric acid is added, and the syrup incinerated. If C be the weight of the ash of the raw syrup, and C' the weight of the ash of the *masse cuite*, the quantity of green syrup per cent. of *masse cuite* will be  $\frac{100 C'}{C}$ , and the percentage of crystals =  $100 - \frac{100 C'}{C}$ .

*Stocktaking and balancing the work from the measurement of the masse cuite and its analysis.*—The density of the *masse cuite* and its saccharimetrical analysis having both been determined, if its volume be also known then the total quantity of sugar produced is also known. This amount of sugar ought to be identical with that contained in the beets treated, which have been weighed and analysed. The *masse cuite* contains the whole of the sugar of the beets minus losses. Its analysis therefore is a safe guide in ascertaining the good or bad working of the factory. The *masse cuite* cannot be weighed except when sugar boxes are used. This is rather unfortunate for stocktaking purposes, because, if the weight and percentage strength of the sugar were known, then all working



data would be available to balance the working accounts of the factory. The large cooling vats cannot be weighed; their measurement and the density of their contents are taken, and the total weight calculated from the far from accurate data thus obtained. But in actual sugar-works practice it is continuously done; in the end the errors correct themselves, and a sufficiently exact statement of each run is obtained. The analysis itself is not free from difficulty; failure to obtain a fair average sample causes numerous errors. Anyhow, the analytical results of the *masse cuite* are those by which not only the amount of sugar extracted from the beet is determined, but also the quality of the sugar is thereby ascertained, and the good or bad working of the factory.

8. *Saline coefficient*.—With good beets the boiling to strike-point results are good, and the ratio of the sugar to the salts contained in the mass is high. This ratio is called the *saline coefficient*, and varies about 20. Above that figure the results are good; below, down to 16, middling; less than 16 denotes bad beets highly charged with saline matter. The *saline coefficient* is therefore a sure guide to warn the manufacturer of the necessity of trying to improve the quality of the *masses cuites*. If this be not done, a poor yield of sugar and much molasses will result. It is, however, impossible to determine exactly the volume of the "firsts" *masse cuite* generally run into large, flat, shallow tanks of great width. A few small fractions of an inch, more or less, produce a great difference in the cubic content. The data got from the value of the "firsts" *masse cuite*, however exactly ascertained, are completely spoiled by the return to it of the turbine drainage liquors. It has been suggested to put these figures to one side, and only to take as a basis the turbinated sugar and the "seconds" *masse cuite*. It is plain that the sum of the sugar contained in the turbinated products and in the drainages is equal to the sugar comprising the "firsts" *masse cuite*, without any addition of turbine drainage. The turbinated sugar is generally weighed, and the "seconds" *masse cuite* is run into vessels deep enough to obviate any error in gauging them. All that has to be done, therefore, is to get a fair average sample of the above products, polarise the turbinated sugar and determine the density of the molasses and the "seconds" *masse cuite*, and all the data are to hand for determining the quantity of sugar contained in the "firsts" *masse cuite*.

# COMPOSITION OF MASSES CUITES AND SYRUPS 197

TABLE XXXVI.—COMPOSITION OF MASSE CUIITE AND SYRUPS OF DIFFERENT DENSITIES (MAUMENÉ).

Weight of the Litre in Grammes.	Syrup of Density 1.400.	Sugar Crystallised.	Total Sugar.	Sugar Per Cent.
1405	1334.75	70.25	1090.378	77.606
1410	1304.325	105.675	1102.562	78.197
1415	1273.50	141.50	1114.812	78.788
1420	1242.415	177.585	1127.155	79.376
1425	1211.25	213.75	1139.480	79.964
1430	1179.825	250.175	1151.885	80.549
1435	1148	287.0	1164.390	81.142
1440	1115.915	324.085	1176.955	81.735
1445	1083.75	361.25	1189.540	82.320
1450	1051.325	398.675	1202.177	82.907
1455	1018.50	436.50	1214.975	83.504
1460	985.415	474.585	1227.705	84.091
1465	952.250	512.755	1240.216	84.676
1470	918.825	551.175	1253.402	85.264
1475	885	590.000	1266.391	85.837
1480	850.915	629.085	1279.431	86.449
1485	816.75	668.25	1292.447	87.032
1490	782.325	707.675	1305.569	87.622
1495	747.5	747.5	1328.862	88.217
1500	712.415	787.585	1332.055	88.804
1505	677.25	827.75	1345.335	89.390
1510	641.825	868.175	1357.571	89.885
1515	606	909	1372.155	90.572
1520	569.915	950.085	1385.651	91.162
1525	533.75	991.25	1399.232	91.753
1530	497.325	1032.675	1412.756	92.337
1535	460.5	1074.45	1426.454	92.927
1540	423.415	1116.585	1440.182	93.519
1545	386.25	1158.75	1453.917	94.108
1550	348.825	1201.175	1467.959	94.696
1555	311	1244	1481.692	95.286
1560	272.915	1287.085	1495.658	95.876
1565	234.75	1330.25	1509.632	96.468
1570	196.825	1373.675	1523.706	97.050
1575	157.5	1417.5	1537.873	97.643
1580	118.415	1461.585	1552.077	98.232
1585	79.25	1505.75	1566.319	98.822
1590	39.825	1550.175	1580.609	99.409
1595	0	1595	1595	100.000

TABLE XXXVII.—DENSITY OF *MASSÉS CUITES* AND SYRUPS AS INDEX TO APPARENT DRY MATTER.

20 % Solution.		<i>Massé Cuite</i> or Syrup.		20 % Solution.		<i>Massé Cuite</i> or Syrup.	
Abridged Density.	Apparent Dry Mater.	Absolute Density.	Apparent Dry Mater.	Abridged Density.	Apparent Dry Mater.	Absolute Density.	Apparent Dry Mater.
A.	B.	C.	D.	A.	B.	C.	D.
5.00	13.15	1.326	65.75	6.30	16.53	1.434	82.65
5.05	13.23	1.329	66.15	6.35	16.67	1.439	83.35
5.10	13.41	1.332	67.05	6.40	16.80	1.444	84.00
5.15	13.55	1.337	67.75	6.45	16.92	1.448	84.60
5.20	13.70	1.341	68.50	6.50	17.05	1.452	85.20
5.25	13.84	1.345	69.20	6.55	17.18	1.457	85.90
5.30	13.99	1.349	69.95	6.60	17.32	1.462	86.60
5.35	14.10	1.354	70.50	6.65	17.46	1.467	87.30
5.40	14.21	1.357	71.05	6.70	17.59	1.471	87.95
5.45	14.33	1.361	71.65	6.75	17.72	1.476	88.60
5.50	14.44	1.366	72.20	6.80	17.84	1.480	89.20
5.55	14.57	1.369	72.85	6.85	17.97	1.485	89.85
5.60	14.70	1.373	73.50	6.90	18.09	1.489	90.45
5.65	14.83	1.377	74.15	6.95	18.22	1.494	91.10
5.70	14.96	1.382	74.80	7.00	18.36	1.499	91.80
5.75	15.09	1.386	75.45	7.05	18.50	1.504	92.50
5.80	15.22	1.390	76.10	7.10	18.63	1.509	93.25
5.85	15.35	1.394	76.75	7.15	18.76	1.514	93.80
5.90	15.47	1.398	77.35	7.20	18.88	1.518	94.40
5.95	15.59	1.402	77.95	7.25	19.02	1.523	95.10
6.00	15.73	1.408	78.60	7.30	19.16	1.529	95.80
6.05	15.87	1.412	79.35	7.35	19.29	1.534	96.45
6.10	16.01	1.416	80.05	7.40	19.42	1.538	97.10
6.15	16.15	1.421	80.75	7.45	19.54	1.543	97.70
6.20	16.27	1.425	81.35	7.50	19.66	1.547	98.30
6.25	16.40	1.430	82.00				

# BEET-SUGAR WORKS ENGINEERING DATA 199

TABLE XXXVIII.—OUTPUT OF PUMPS IN LITRES PER HOUR REQUIRED FOR RETURNS FROM HEATING, MULTIPLE-EFFECT, AND STRIKE PANS, AND TO SEND THEM INTO THE BOILERS, OR, IF DESIRED, INTO THE SPECIAL RESERVOIR (*BALLON DES RETOURS*) (CAMBIER).

A.	B.	C.	D.	E.	F.	G.
1200	707-30	1362	2241-80	4883-60	500-00	150-00
1600	944-80	1816	3248-40	6496-80	665-60	195-20
2000	1180-30	2270	4068-00	8136-00	832-00	244-00
2400	1414-60	2724	4883-60	9767-20	1000-00	300-00
3200	1888-00	3632	6496-80	12992-60	1331-20	390-40
4000	2360-00	4540	8136-00	16272-60	1664-00	488-00
4800	2829-20	5448	9767-20	19534-40	2000-00	600-00
5600	3302-60	6356	11360-40	22759-80	2331-00	690-40
6400	3774-60	7264	13019-60	26089-20	2264-00	788-00

A, Hectolitres of juice treated in 24 hours. B, C, D, E, F, G, output of pumps in litres per hour: B, from diffusion. C, from carbonatation. D, from No. 1 of triple-effect. E, from Nos. 2 and 3. F, from "firsts" strike pan. G, from "seconds" strike pan. E, returns to reservoir. All others to the boilers.

TABLE XXXIX.—STEAM CONSUMED PER TON OF BEETS IN BEET-SUGAR FACTORY WORKING 300 TONS OF BEETS IN 24 HOURS.

Operation.	A.	B.	C.	D.	E.	F.
Diffusion . . .	107-69	...	...	...	...	107-69
Carbonatation . .	223-73	...	...	...	...	223-73
Evaporation . . .	426-77	...	...	...	...	426-77
Heating syrups, boiling "firsts" . .	85-24	...	...	...	...	85-24
Boiling "seconds" .	25-68	...	...	...	...	25-68
Engines . . . .	...	584-211	426-77	25-44	129-64	129-84
Engines <sup>1</sup> . . . .	...	...	...	...	...	25-44
	869-11					1024-39

Kilogrammes of steam expended: A, in heating and evaporating. B, by engines, partly restored to evaporation. C, restored to evaporation. D, excess of consumption over evaporation. E, actually expended without restitution. F, total expended on heating, evaporation, and motive power.

<sup>1</sup> Expended in motors without restitution.

TABLE XL.—EXPENDITURE OF STEAM BY EACH ENGINE IN A BREIT-SUGAR WORKS TREATING 300 TONS OF BEETS IN 24 HOURS.

	A.	B.	C.	D.	E.
Diffusion . . .	500	1 m. 600	1,130,688	904,550	2,951 k. 727
Sludge pumps . .	230	1 m. 000	149,400	119,520	389 186
Gas (CO <sub>2</sub> ) . . .	350	1 m. 120	987,878	810,302	1,012 577
Triple-effect . .	350	1 m. 280	443,335	354,668	1,158 352
Masse cuite . .	320	1 m. 190	344,519	275,615	899 896
Turbines and pump	320	1 m. 300	376,365	301,092	982 525
Feeding . . .	200	0 m. 525	59,346	47,476	154 923
			Total . .		7,548 k. 625

A, diameter of pistons in mm. B, speed of pistons per second. C, volume generated hourly by pistons in litres. D, volume of steam in litres admitted hourly—admission 80 %. E, weight of steam consumed hourly.

TABLE XLII.—STEAM CONSUMED PER HECTOLITRE OF JUICE IN THE DIFFERENT BRANCHES OF A BREIT-SUGAR WORKS WORKING 300 TONS IN 24 HOURS. JUICE OBTAINED PER TON, 1,505 LITRES.

	A.	B.	C.	D.	E.	F.
	Kg.	K.	K.	K.	K.	Kg.
Diffusion . . .	7-155	...	...	...	...	7-155
Carbonatation . .	14-800	...	...	...	...	14-800
Triple-effect . .	28-356	...	...	...	...	28-356
Heating syrups, boiling "firsts"	5-663	...	...	...	...	5-663
Boiling "seconds".	1-706	...	...	...	...	1-706
Engines . . .	...	88-610	28-356	1-690	8-620	1-690
Engines . . .	...	...	...	...	...	8-620
	57-680					67-990

Kilogrammes of steam expended: A, in heating triple-effect. B, by engines, partly restored to evaporation. C, kilogrammes of steam restored by engines to evaporation. D, excess of consumption over evaporation. E, kilogrammes of steam actually expended without restitution. F, total steam expended on heating, evaporation and motive power.

TABLE XLII.—WORKING DATA AND RESULTS OF VACUUM STRIKE PAN.

A.	B.	C.	D.	E.	A.	B.	C.	D.	E.
15	3.55	254.61	71.72	42.28	22	1.91	91.51	47.91	78.09
16	3.11	210.81	67.78	48.22	23	1.78	78.08	43.87	84.13
17	2.76	175.92	63.78	54.27	24	1.61	60.89	37.82	93.18
18	2.49	148.93	59.81	60.19	25	1.56	56.01	35.90	96.10
19	2.38	137.61	57.81	63.19	26	1.46	46.44	31.80	102.20
20	2.26	126.02	55.75	66.25	27	1.38	38.47	27.87	108.13
21	2.08	107.86	51.85	72.15	28	1.31	31.26	23.86	114.14

A, degree Baumé at exit from multiple-effect. B, quantity of syrup necessary per hectolitre of *masse cuite*. C, amount of water to evaporate to obtain a hectolitre of *masse cuite*. D, water evaporated per hectolitre of syrup to transform it into *masse cuite*. E, *masse cuite* obtained per hectolitre of syrup.

## CHAPTER VI.

### TURBINING OF BEET-SUGAR MASSES CUITES.

*TURBINES.* The magnitude of the centrifugal force in turbines.—In the case of a point revolving in a circle, of which the radius = R and the time of revolution = T, the magnitude of the centrifugal force is  $\frac{4T^2R}{T^2}$ .

Supposing R = 2 feet, and that the particle makes 10 revolutions per second, the magnitude of the force is therefore  $4 \times \left(\frac{22}{7}\right)^2 \times 2 \times 100$ . This quantity, when calculated out, gives 7884. But the force of gravity is 32; therefore the ratio of the centrifugal force to gravity is  $\frac{7884}{32} = 246$ . The centrifugal force is therefore 246 times greater than gravity. Each particle of water, therefore, between the crystals of sugar is expelled by a force 246 times greater than its own weight. The great advantage in separating liquid molasses from crystalline sugar by centrifugal force lies in the fact that each atom of water is expelled by a force acting on itself directly, and not transmitted to it by the pressure of and through the intermediary of the surrounding particles. If it were attempted to express molasses from sugar by hydraulic pressure, as oil is expressed from seeds, the particles of sugar on the outside of the cake would be crushed, but the pressure would be but very imperfectly transmitted to the interior. Unless the crystals were so compressed and crushed together as to annihilate all the intervening interstices, the water would not be removed from the bulk of the mass. Such a result, it is needless to state, could not be obtained by pressure without crushing and destroying the beauty of the crystals beyond all recognition. But, when centrifugal force comes into play, its action is altogether different: the crystals, though as tightly and closely packed together as possible, are quite uninjured; their crystalline symmetry remains intact, whilst at the same time numerous inter-

stices intervene between the crystals, and out of these the water is ejected by the centrifugal impetus of its own inertia.

*The history of the sugar turbine.*—The idea of applying the principle of the centrifugal machine—the French “turbine”—appears to have been suggested by the turbines previously existing in establishments for bleaching, etc., of textile fabrics. Moisture had been thus expelled from fabrics long before any one ever thought of using turbines in sugar works. It is not known exactly when or in what country they were first used for sugar. In the year 1850 no less than thirty-six patents were taken out in Great Britain alone for the employment of new and improved forms of sugar turbines. The turbine has been aptly described as a wire gauze cup, strongly fortified with wire and with bars, mounted on a vertical pivot, and capable of revolving at as high a velocity as 1,500 revolutions a minute; it, in fact, consists of a vertical cylindrical open-topped drum (with a vertical axis), 2 to 2½ feet in diameter, the side of which consists of wire gauze strengthened as described. The bottom is closed, and a conical portion surrounds the spindle or axis of rotation. The whole is enclosed in a cylindrical cast-iron case to match, having in its bottom an arrangement on which the axis or spindle rests.

*Method and principles of working.*—It may be driven from above by friction cones (Fig. 74), or from underneath the drum by belting (Fig. 64).

*Speed.*—In beet-sugar factories the speed is generally 400 to 600 revolutions a minute. Whilst revolving at this speed, if a certain weight of *masse cuite* be run into it—say 1 cwt.—the whole mass is projected against the side of the basket by centrifugal force. As the side consists of tight wire gauze the molasses pass through, whilst the crystals remain behind in the basket. By cleansing the crystals with water, pure syrup, or steam (Fig. 71), or even by a blast of moist air, the final portions of the molasses which soiled them are eliminated, and white granulated sugar is obtained.

*Acceleration of speed with different grades of sugar.*—The wall of sugar in basket should be vertical. Molasses are then uniformly eliminated and cleansing liquor purges uniformly. With full charge of good sugar, from starting to full speed should not be more than 1½ to 2 minutes; but all individual centrifugals working in battery should be uniformly accelerated collectively, a desideratum only perfectly attained by water-driven centrifugals, constructed with two or more water jets. Used conjunctly, or singly, rapid or slow acceleration is attained, a regulator automatically closing those jets rendered unnecessary by the attainment of full speed. Rapid acceleration so packs small grained or gummy sugar against the lining that the molasses cannot penetrate through it. Speed should be rapidly accelerated when curing good, free, easy-drying sugars and very



slowly with bad sugars, and in any case slow enough to hinder the molasses flowing to inside of sugar wall.

*Types of turbines.*—Turbines are of three types (1) The French turbines, driven from above (Fig. 74); (2) the Austrian turbines driven from underneath (Fig. 64) (Fesca's type); (3) suspended turbines on what is known as the Weston type, i.e., driven from above and discharging underneath.

(1) *French type of turbine.*—The French turbine, the axis of which is held by the two ends, is more stable and does more work.

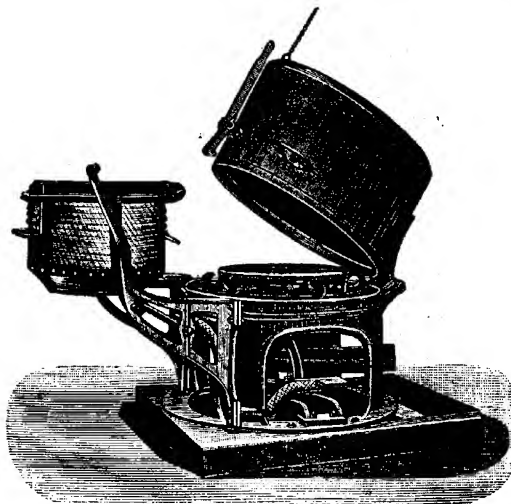


FIG. 64.—Turbine driven from underneath (HANROLD).

But the driving of it from above (Fig. 74) involves a cumbersome arcade supported on the framework of the machine. (2) *Austrian type of turbine.*—The turbine driven from underneath allows the drum, which is also called the basket, to be got at in any part of its surface, which is a great advantage in filling. But the axis is held in two near points—(1) at the bottom, and (2) near the centre—in such a way that the latter is a false door. The result is that a nutation motion is produced on starting, which Fesca has corrected by a special regulator in the cone present in the centre of every turbine to regulate the spreading out of the *masse cuite*. The French

makers of this latter kind of turbine have partially corrected this inherent defect by placing the second point of attachment of the axis at the centre of the cone so that the centre of gravity of the turbine is between the two fixed points. They thus dispense with Fesca's regulator. Nevertheless, the excellent qualities of the French turbine (Fig. 74) cause it to be often preferred, in spite of its defects, to the Austrian (Fig. 64), which is only utilised when there is no other resource, particularly in the special type of turbines for refined sugar, such as the Fremaux turbine, which necessitates the basket being quite open. The turbine shown in Fig. 64 may be

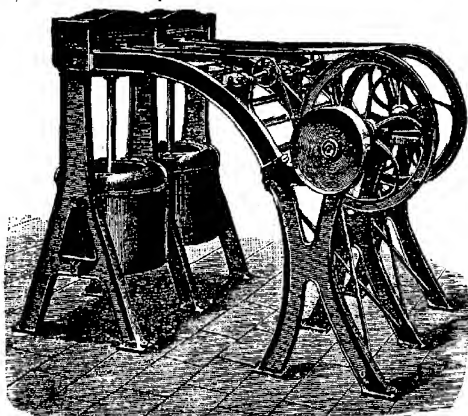


FIG. 65.—Weston's suspended turbines (WATSON, LAIDLAW & Co.).<sup>1</sup>

made to do the work of two; whilst one drum is being discharged and refilled, the other is being rotated. (3) *Weston's turbine*.—There is a third kind of turbine, of American origin, formerly used more especially in cane-sugar factories, although now also extensively used in beet-sugar factories as well. This is Weston's turbine, (Fig. 65). The basket is hung at the lower end of a vertical axis, which is only held by a single point above at the other end of the axis at the centre of the driving pulley, in such a way that the belt pulls on the point of suspension without interfering with the vertical position of the axis. These turbines have therefore been constructed so as to be emptied from below, the central cone

<sup>1</sup> Inadvertently credited in first edition to Cambray. The author's apologies have already been tendered to Messrs. Watson, Laidlaw & Co.

being capable of being displaced vertically, leaving a hole wide open through which the sugar is pushed, without touching it by hand, by means of a small shovel. The sugar thus escapes being soiled by the not over-clean hands of the workmen.

*French form of turbines discharging underneath.*—The discharging of turbines from below, having some advantages, has also been effected in the case of the French turbines, by leaving an opening in the bottom of the drum, which closes with a sliding door, which corresponds with a funnel in the cast-iron cover in which every turbine is enclosed, and in which the turbinized molasses collect.

*A great field open for continuous turbines.*—A continuous turbine (Fig. 66) has recently been invented into which the *masse cuite* regularly flows without stopping, and which only discharges turbinized sugar. The principle of this machine is, that the drum, instead of

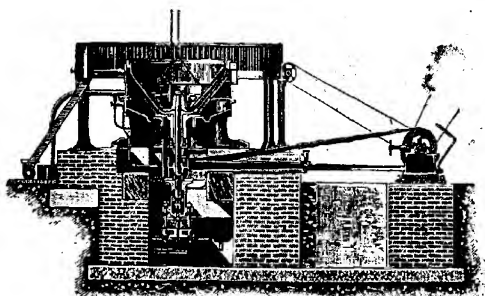


Fig. 66.—Continuous turbine, Szezentowsky and Piatkowsky's system (GAIL).

being a cylinder, is a reversed cone. It is fed from the bottom of the cone, and the centrifugal force causes the sugar to ascend up the gradually widening conical sides. At first this machine only produced yellow sugar. Whiter sugars are now got. This invention is perhaps rendered even more interesting still by the fact that there is a great future in store for continuous machines, and there is, especially in the case of the turbine, a great advantage to be gained by automatic working which has not hitherto been accomplished. Watson, Laidlaw & Co. also make a continuous form of turbine on Robb's patent.

*Plan of the turbine room.*—The turbinizing of the *masse cuite* requires a well-planned installation, so as to be done economically; failing which the manual labour is considerable, because the least flaw in the arrangements stops all the work, which has then to be done by hand.

*The old routine.*—The work of the turbine house was formerly carried on as follows: The *masse cuite* collected in the flat tank being very hard, was removed with pick and shovel by men standing in the tank. This was a very laborious job. At the side of the tank was a mixer, a kind of toothed mill surmounted by a hopper, into which the men threw the *masse cuite* by shovelfuls. The mixer rendered the mass homogeneous by a jet of molasses which continuously trickled into the hopper to moisten the mass. Under the mixer was a door, through which its contents were dis-

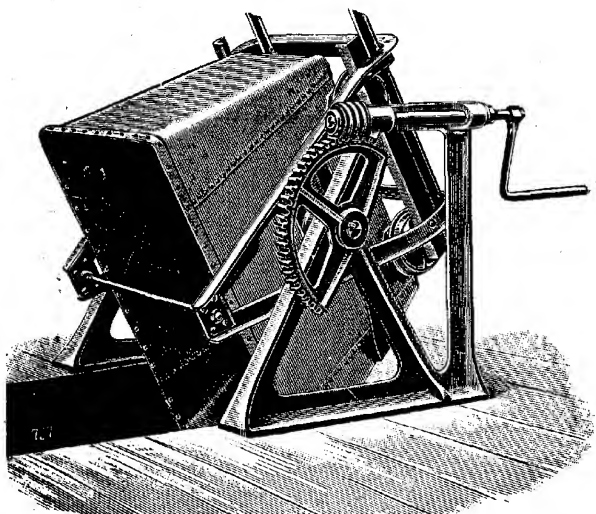


FIG. 67.—Concrete sugar truck in tipping winch, tilted, discharging contents into sugar breaker feeding-trough (WATSON, LAIDLAW & Co.).

charged into a basin placed underneath, which held enough to charge one turbine. Two men seized this basin, emptied it into the turbine, replaced it under the mixer to be filled to charge another turbine, and so on.

*The recent and more or less automatic methods.*—The whole, or the greater part, of this primitive method of working has been replaced by something more automatic. Various forms of sugar trucks, etc., are used (1) to cool the *masse cuite* and (2) convey it to the pug mill or mixer. Figs. 61-63 and 67, 68 and 69 will require no explanation.

If the flat tank be retained, men have still to shovel the mass. But instead of shovelling it directly into the mixer, they throw it into a trough placed within reach, in the bottom of which a helix revolves. The molasses for moistening falls continuously and uniformly into this trough. The helix conveys the mass into the mixer, and from the mixer it falls into another helix or inclined Archimedean screw, which raises it into a feeding-trough with stirrers and driving gear placed horizontally behind the long line of turbines and extending all its length. The revolving shaft, fitted with blades, keeps the mixture uniform. In front of each turbine a door is inserted in the trough, opening into a chute, which conveys the moistened mass

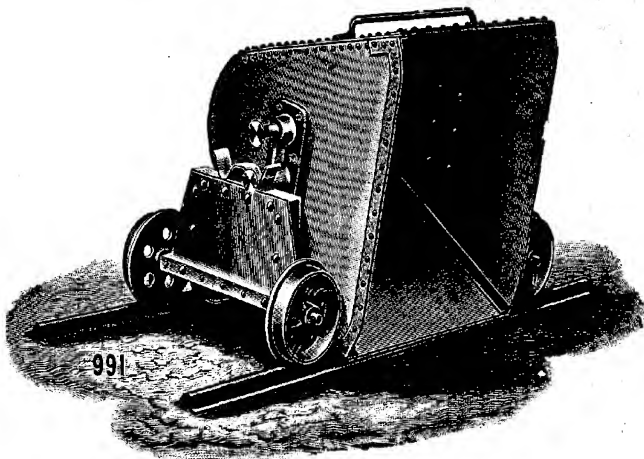


FIG. 68.—Concrete lateral tilting sugar truck on rails (WATSON, LAIDLAW & Co.).

into the basket of the turbine. When the turbine is charged, the door is closed. But charging thus is irregular. A small measuring tank was then placed between the trough and each turbine, which was filled by opening the door, and closing it when it was perfectly full. It was emptied into the turbine through another door. A still better arrangement was effected when *the two doors were made to work by a single pull with one arm of a lever, the one door only opening when the other door is closed.*

The French turbines shown in Fig. 74 are charged automatically from measuring vessels, the arrangements for which are partially shown in the illustration.

## TURBINING OF BEET-SUGAR MASSES CUITES 209

*The helical systems spoil the grain of the sugar.*—All these helical systems work very well, but are blamed for spoiling the grain.

When the plan of the place permits of it, it is preferred to elevate the cooling tank, so that the mixer discharges directly into the distributing tank, with its small measuring tanks. This method works well.

*Charging by overhead trucks.*—Where an under-driven turbine is used, entirely open above, the turbines are filled from small trucks opening underneath (Fig. 69) and containing the correct charge of one turbine. These trucks run on rails suspended above the turbines; they are filled directly under the mixer, and are then

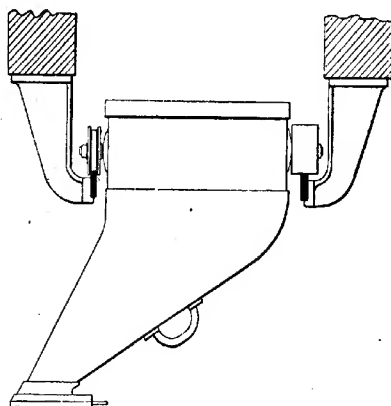


FIG. 69.—Overhead truck running on rails for charging turbines—(WATSON, LAIDLAW & Co.).

brought above the turbines, and their contents are simultaneously emptied into the turbine. This system of trucks is indeed the only one to be recommended when there is a long row of turbines; the helices do not work regularly in such a case.

These overhead trucks are also used where the sugar has to be brought some distance to the centrifugals, or where it cannot conveniently be discharged direct from a mixer or pug mill. The overhead railway is arranged, if possible, so that the truck may make a complete circuit. Two standard forms of trucks are made—one single and the other double—the latter having two compartments, each compartment being capable of holding a charge for one

machine. The trucks are of strong galvanised iron, and have swivel wheels to run on rails either straight or curved, and are also fitted with gun-metal sluice valves. The overhead railways are usually of a very simple but substantial construction. The hangers to which the rails are attached may be fixed to timber beams as shown, or otherwise, as may be convenient. Trucks with side openings have also been built for use with French turbines, and they answer well, although they would not be so convenient with the under-driven turbines. When it is customary to run the *masse cuite* into the small sugar boxes (Fig. 70), the place for the mixer can be chosen; it can be placed on a higher floor to the turbines, and trucks may be used without difficulty.

*Discharging the sugar boxes by compressed air.*—After the sugar has cooled and set, the boxes are taken to the sugar breaker. In emptying these sugar boxes into the mixer, care is taken to leave a small hole in the bottom of the box, closed by a screw stopper. The box is brought full above the mixer, and, being held by its wheeled support, it is turned upside down (*i.e.*, tilted), the cork is unscrewed, and through this opening (Fig. 70) a compressed air-pipe is introduced, which presses behind the mass and forces the whole block right out of the box. This conical block is seized by the teeth of the mixer and thoroughly incorporated with a little molasses and brought into a suitable condition for charging the turbines. The work is done well and very rapidly. The nozzle, which fits into the hole in the bottom of the sugar box, is connected with a small air compression pump by a length of indiarubber tubing (Fig. 70).

*The crystallisation in motion masse cuite receiver and its numerous advantages.*—The well-mixed boiled mass, cooled to a nicety in this vessel (pp. 186-190), is collected with the greatest of care in the trucks for charging the turbines through a door fixed in the bottom of the mixer. Here (1) workmen are no longer required to extract the mass in the tank; (2) no more destruction of crystals; (3) easy and correct working; (4) a large number of the turbine staff of hands are dispensed with; (5) great cleanliness in the turbine house—everything which one can desire of the best for this very delicate operation. Of all equipments, this presents the greatest number of advantages; only, the vacuum pans must be elevated one story further up, which, however, does not cause much trouble.

*General method of working turbines.*—When charged, the turbine is generally first started by hand, then the driving strap is put on near the cones, according to the model of machine, and it is allowed to turn during the number of minutes which correspond with the degree of purity which it is desired to obtain in the crystals.

*Different grades produced by different treatment in the turbine.*—It may be necessary to make extra-white sugar, as in Russia, or as in France—cube sugar, white sugar, yellow sugar or brown sugar,

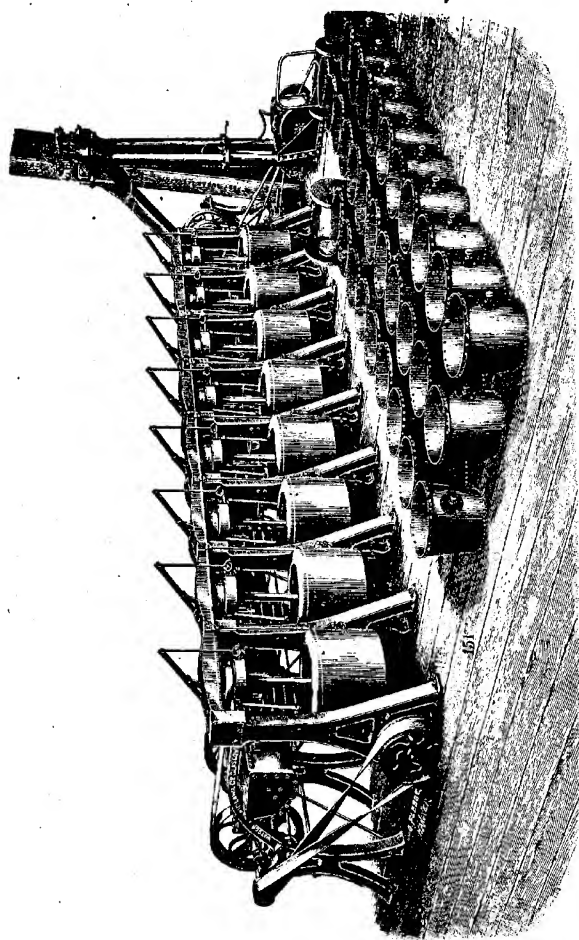


FIG. 70.—Set of eight 30-inch (762  $\text{m}/\text{m}$ ) machines, with independent framing, countershaft, friction pulleys, steaming-out cocks, molasses gutters, mixer, sugar breaker, pug mill, wet sugar elevator, screw conveyor, dry sugar elevator, air compressor, sugar boxes and carriages (WATSON, LAIDLAW & CO.).



according to the custom of the market and the commercial skill of the manufacturer, all of which qualities require a special turn of the wrist. In making extra-white sugar the turbine must be kept

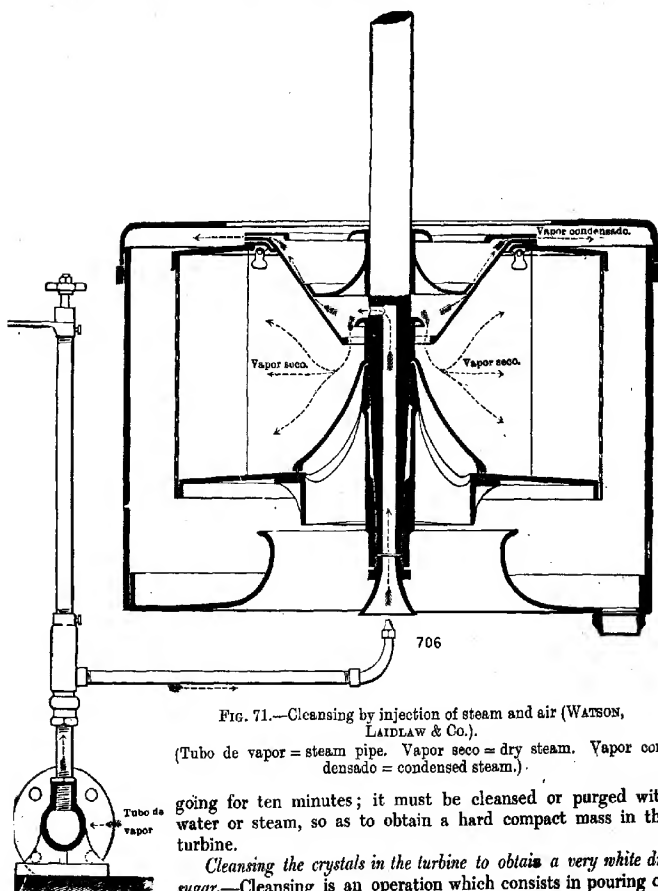


FIG. 71.—Cleansing by injection of steam and air (WATSON, LAIDLAW & Co.).

(Tubo de vapor = steam pipe. Vapor seco = dry steam. Vapor condensado = condensed steam.)

going for ten minutes; it must be cleansed or purged with water or steam, so as to obtain a hard compact mass in the turbine.

*Cleansing the crystals in the turbine to obtain a very white dry sugar.*—Cleansing is an operation which consists in pouring on

the mass in motion a certain quantity of pure sugar liquor or water. This liquid displaces the molasses which the centrifugal force cannot detach from the grain. If the cleansing liquor be a sugar syrup, it takes the place of the molasses; if it be water it dissolves a certain quantity of sugar and acts like the syrup cleanser, which is nothing more nor less than sugar dissolved in water. Cleansing by steam acts in the first place like water, because a portion of the water condenses between the crystals; but when the sugar has reached a certain temperature in consequence of this condensation, the steam, no longer condensing, dries the crystals and frees them from the very fluid, hot, cleansing liquor covering them. There is then found in the turbine a very white and very dry sugar.

*Method of cleansing to get an ordinary white sugar.*—If an ordinary white sugar be required, a solution of sugar is used less pure than that employed in the first instance. That flowing from the turbine at the time of steaming, as a yellow liquid, infinitely purer than the primitive molasses, is used. In that case, cleansing is done by steam. Four to five minutes suffice to obtain this product.

*Ordinary yellow sugar.*—To make ordinary yellow sugar, they turn three or four minutes. The time and nature of the cleansing liquor depend on the percentage test desired. If no cleansing liquor be added, brown sugars, giving a low percentage test and still containing a certain amount of molasses, are produced.

*Cleansing by steam injector.*—Steam, in the cleansing of the crystals, dissolves much sugar; a mixture of air and steam by means of a steam injector, bringing a certain amount of air in its train has been substituted. This answers well, and is more economical than steam alone. Hot moist air has also been used. But these systems were for some time not so general; they did not appear to have any great advantage over simple steam.

But the use of steam, or steam and air, for purifying sugar in the turbine has greatly extended of recent years, and will, no doubt, be still more generally adopted as its advantages in many classes of sugar become more widely known. The advantages to be gained, however, depend entirely upon the way in which the application of the steam apparatus to the centrifugal is carried out—especially upon the provision made for the removal of condensed vapour without permitting it to come in contact with the sugar. In the apparatus shown above, the steam and air are introduced into the baskets from below, through a hole bored in the spindle. The surplus moisture is removed by centrifugal action, causing it to flow up the inner surface of the cone, as shown by the arrows, and escape at the lip into the outer case. The steam which passes into the interior of the basket condenses on the surface of the sugar, and is thereafter driven through the wall of sugar by centrifugal action. This method has been found very efficient, and has been largely adopted. Can be made to suit any size of centrifugal.

*"Firsts" sugars and "firsts" syrups.*—The molasses which soiled the crystals of sugar in the *masse cuite*, pass, as already mentioned,

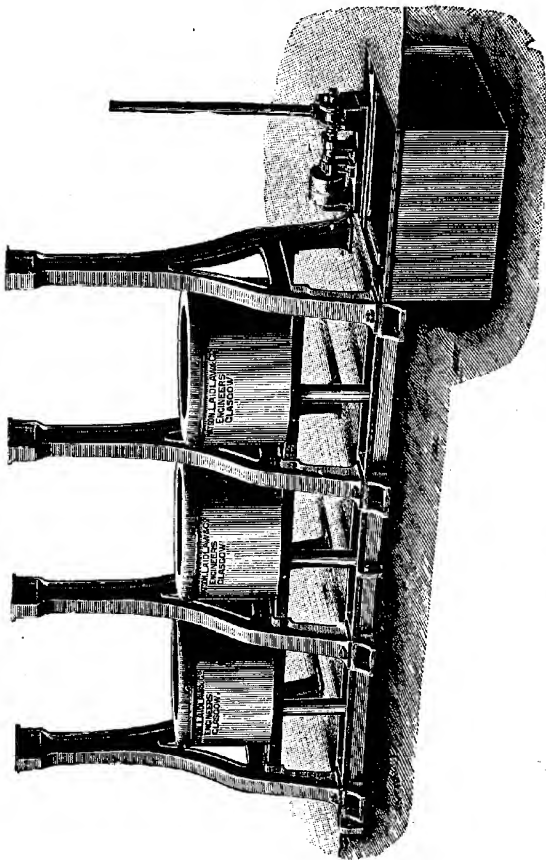


Fig. 72.—Molasses gutters and tanks for Weston's centrifugal.

through the wire gauze of the turbine, are projected against the side of the cover, and issue through a hole made at the bottom of

this cover, constituting what is called the syrup from the "firsts" *masse cuite*; the sugar obtained being called "firsts" sugar.

*The quality of syrup improves from beginning to end of turbinizing process.*—The quality of the syrup varies with the progress of the turbinizing. It is very impure at the outset, since it consists of molasses, but it becomes very fine towards the end. Formerly no heed was taken of this difference, and all the drainings flowed into the same tank—a most wasteful method. But, later on, these drainings were separated. The first only went to the common tank. The second, of the same purity as sugar factory syrup, and purer still towards the end of the process, was sent back to the strike pan to be boiled, like ordinary syrup. The two are separated by providing the exit with a movable gutter, which the workman directs either into the impure drainage trough or into a parallel one for the pure drainings. This change may even be done automatically at the same time that the steam valve is opened for clarifying, so that the workman does not forget it; for everything which helps the workman and does not leave the work at his mercy prevents errors and mistakes. During the season 1899-1900 thirty-five factories in France did not return the drainage syrups or "égouts" for further treatment, sixty-four returned them to the raw juice or to the carbonatation, whilst 267 returned them to the vacuum pan. To ensure good and economical working, these drainage liquors are analysed, and those which are pure enough are transferred to the "firsts" vacuum pan, whilst the remainder are sent to the "seconds" pan. The drainage liquors from the "seconds" are almost always rich enough to be reboiled. This is the case when the yield of "firsts" and "seconds" is not very high. With good yields from the "firsts" and "seconds" turbines, the drainage from the "seconds" are quite poor enough to be sold as molasses. *The saline coefficient of molasses, i.e., its sugar divided by its ash, (p. 195) being about five, when this figure is reached there is little chance of getting crystallisable "thirds".*

*The boiling of low or poor quality products.—Concentrating and crystallising the syrup from the "firsts".*—The syrup or drainage from the "firsts" as it flows from the turbine is still capable, under favourable conditions, of producing crystals. This molasses, which moistens the grains in the *masse cuite*, is still very rich, and it becomes richer still by the melting of the sugar during cleansing. It is necessary to filter it to remove any solid and mucilaginous impurities which it may contain, and then to evaporate it to bring it to the strike point, and then to abandon it in tanks at a temperature of 30° to 40° C. (86° to 104° F.), to enable it to crystallise at the end of a few days. The boiling of the "seconds" is generally done in a special vacuum pan used for this purpose. Sometimes the "seconds" are boiled in the same pan as the "firsts," between two boilings

when there is time. But it is better to use a special pan for this work. The "seconds" *masse cuite* is run into tanks of the same capacity as the vacuum pan. They are arranged in a row, and are sufficient in number to contain the work of two months, the maximum time required for their crystallisation.

*Variations in quality and consistency.*—The crystalline mass is sometimes very hard if the "seconds" are rich, sometimes mushy if they are poor. That depends on the yield of the "firsts," because, if the "firsts" are abundant, as they should be, there will be but little "seconds". These differences originate in general in the good or bad management of the factory, and are also due sometimes to the quality of the beets which have been wrought. The "seconds" *masse cuite* crystallised in the tanks is turbinéd, and gives "seconds" sugars, which are yellow. The drainage from the turbines ought to be reboiled, again run into the crystallising tank, and afterwards turbinéd. It yields "thirds". It is rarely that "fourths" (Table XLV.) are made in beet-sugar manufacture. The time required for the crystallisation of the "thirds" is much longer than for the "seconds". It requires at least six months. They are therefore very often not turbinéd until the factory starts work again in the following season, so as not to resume this work in the summer, bringing workers back expressly and the whole of the staff of the factory.

*The fill-houses.*—The "firsts" and "seconds" tanks are contained in vast buildings called fill-houses. These fill-houses occupy the whole of the ground floor of these buildings, and the upper floor is generally occupied by the sugar warehouses, which should be correspondingly large. The fill-houses ought to be continually kept at a temperature of 40° C. (104° F.). This temperature is obtained either by steam heating or calorifers, or by kinds of *brasero* (braziers), which burn night and day in the rooms; calorifers heated from the outside being the most practical. However, during manufacturing, steam heat is generally used, and there is also great economy in utilising the steam from No. 1 or No. 2 vessel of the triple-effect plant to get the temperature required for the heating of the "seconds".

*Manufacture of brown sugar.*—The more pure the *masse cuite* is, and the more impure the syrup separated from it centrifugally, the greater is its yield in sugar. In the old system of working (large or small boiling vessels) it is generally impossible to lower the purity of the discharge below 75 per cent. of real sugar. But since *crystallisation in motion*, etc. (see pp. 186-190, and Figs. 57-60) have come into favour, this purity may, within certain limits, be lowered at will. All that has to be done is to sufficiently concentrate the *masse cuite*, and then to keep it in motion a certain time at a given temperature. There is thus obtained much greater yields of "firsts". But up to what point is this increase remunerative? To elucidate this question Dr. Claassen has prepared two tables.

# TURBINING OF BEET-SUGAR *MASSES CUITES* 217

Table XLIII. shows the influence of the increasing purity of the boiled mass on the yield when the discharge is of constant purity; whilst Table XLIV. shows the variations in yield with constant purity of the boiled mass, but with variable purity of the discharge. Table XLIII. has been calculated on the supposition of 6 per cent. of moisture in the *masse cuite*, say 94 per cent. of dry matter. The sugar extracted is supposed to be 96 per cent. polarisation, 2 per cent. of water, 2 per cent. of non-saccharine, 98 per cent. of purity; the discharge has a purity of 72. The amount of yield is determined according to Hulla's formula—

$$\text{Sugar per cent.} = \frac{S^m C^p - E^p}{S^m (S^p - E^p)}$$

In this formula  $C^m$  = dry matter per cent. of boiled mass;  $S^m$  = dry matter per cent. of turbinéd sugar;  $C^p$ ,  $S^p$ ,  $E^p$  = the quotients of purity respectively of the *masse cuite*, turbinéd sugar, and the green discharge (quotient of purity = ratio of sugar to total dry matter).

TABLE XLIII.—INFLUENCE OF PURITY OF THE *MASSE CUITE* ON THE YIELD IN SUGAR WHEN THE DISCHARGE SYRUP HAS A CONSTANT PURITY OF 72.

A.	B.	C.	D.	A.	B.	C.	D.
94	88	59.0	3.7	94	92	73.8	3.7
94	89	62.7	3.7	94	93	77.5	3.7
94	90	66.1	3.7	94	94	81.2	3.7
94	91	70.1	3.7	94	95	84.9	3.7

A, dry matter in *masse cuite*. B, purity of *masse cuite*. C, yield of raw sugar per cent. of *masse cuite*. D, increase of yield per each cent. of purity.

In Table XLIV. the *masse cuite* has been taken as containing 94 per cent. of dry matter and 91 per cent. of purity, sugar at 98 per cent. dry matter and 98 per cent. of purity; the discharged syrup decreasing in purity from 75 to 66.

TABLE XLIV.—INFLUENCE OF THE PURITY OF THE DISCHARGE ON THE YIELD IN SUGAR OF THE BOILED MASS WHEN THE PURITY OF THE LATTER IS CONSTANT.

A.	B.	C.	A.	B.	C.
75	66.7	...	70	71.9	0.9
74	67.9	1.2	69	72.7	0.8
73	69.1	1.2	68	73.5	0.8
72	70.1	1.0	67	74.2	0.7
71	71.0	0.9	66	74.9	0.7

A, purity of discharged syrup. B, yield per cent. of *masse cuite* in raw sugar. C, increase of yield by diminution in purity of 1 per cent.

According to Table XLIII., for an increase of 1 per cent. on the purity of the boiled mass, other conditions being granted, the yield of the *masse cuite* increases 3·7 per cent. According to Table XLIV., a lowering of the purity in the discharged syrup of 1 per cent. only leads to an increased yield of 1·2 to 0·7 per cent. of raw sugar in the *masse cuite*. This is easily explained, since the weight of the discharged syrup hardly represents one-third of the *masse cuite*. The increase in purity of the *masse cuite* should therefore be the best method of obtaining greater yields. Unfortunately this method is not practicable, supposing always that we employ the same methods as in the past—diffusion, defecation and saturation. The purity of the *masse cuite* depends therefore more especially on the beetroot, and thus is a factor which varies from year to year. But discharge syrups of a low degree of purity may always be obtained by *crystallisation in motion*. It remains to be seen up to what point it is advantageous to lower this coefficient. Two principal factors are to be considered—the duration of the agitation, and the quality of the product obtained. Experience has shown that it is possible in a short time, generally from twelve to twenty-four hours, to reduce the purity of the discharge from 75 to about 70, but that each unit gained below this limit requires a greater and greater loss of time, and consequently involves a correspondingly increased extension of plant. As far as the quality of the sugar is concerned, the product of *crystallisation in motion* is better than that of turbinizing, after allowing the boiled mass to stand in the crystallising tanks. The sugar of *crystallisation in motion* consists of larger and more regular crystals, and it contains a more fluid syrup. If the agitation be prolonged so as to lower the quotient of purity of the discharged syrup below 70, more and more sugar will crystallise, which in ordinary working would have been obtained in the “seconds”. Now this sugar is not so white as that which crystallises during *boiling in vacuo*, and at the beginning of the agitation of the *masse cuite* in the crystallising vessel, and it is so much greyer or browner in tint the lower the purity of the surrounding syrup has been reduced. Thus, whilst the nucleus of the crystals consists of sugar absolutely white, the outside layers are coloured to a greater and greater extent. Naturally, the coloration depends greatly on the shade of the concentrated juice from which the boiled mass was produced. Bad dark juices, especially those resulting from unripe or diseased beet-roots, often yield crystals absolutely deprived of whiteness. But, even in the case of very white syrups, the sugar which crystallises from discharges of less than a purity of 70 is not white. Now, as the first condition for a good “firsts” sugar is to produce, by refining, crystals as white as possible, it is evident that in the manufacture of these sugars it is necessary to avoid prolonging the crystallisation in motion for too great a time. A purity of 70 to 72 ought therefore

to be regarded as normal in the working of "firsts" *masses cuites* by crystallisation in motion, so much the more as each unit of purity corresponds to 0.7 to 0.8 per cent. of increased yield—a gain which would not appear to be in proportion to the loss of time and diminution of quality in the product. If it be desired to apply crystallisation in motion to inferior quality syrups, it would be preferable to work up these syrups by themselves with remelted low quality sugars. Claassen (p. 217) states that Hulla's formula is perfectly applicable to crystallisation in motion. The syrup added to the *masse cuite* must also be taken into consideration as an indifferent body, having, however, two conditions to fulfil. The first condition is to mix with the primitive syrup, so as to prevent the latter becoming too concentrated, and thus give birth to fine crystals instead of enlarging the crystals already formed. The second is that the syrup added fixes any water which may be in excess for the crystallisation of the syrup of the boiled mass, and would finally be opposed to the complete crystallisation of the latter, except for the purity of the added syrup. In fact, the added syrup has no influence on the increase of yield of the *masse cuite*, and may therefore be left out of account. In working, by adding syrup towards the end of the boiling, according to Bock's process, the composition of the *masse cuite* is unknown. In that case the purity of the mass may be determined from that of the concentrated juice of the beet-root as identical with the latter, if the slight loss of sugar due to heating be deducted. The quantity of water in the boiled mass still remains to be ascertained. But it may be taken in all cases as 5 or 6 per cent., according to what it formerly was. With these elements of the composition of the sugar, as well as the purity of the syrup, being easily determined directly, Hulla's formula may be applied.

*Example.*—Average of one week: purity of syrup added to the boiling, 92; composition of the sugar, 96 per cent. polarisation, 2 per cent. water, and 98 purity; purity of the turbinéd syrup, 72; water in the *masse cuite*, 5 per cent.

The yield in raw sugar of the *masse cuite* in weight—

$$\frac{95(92 - 72)}{98(98 - 72)} 100 = 74.5 \text{ per cent.}$$

*Transferring the turbinéd sugar to the sugar granary.*—When it is thought that the *masse cuite* has been sufficiently turbinéd the machine is stopped, and the sugar collected, either by filling it into sacks or by letting it drop under the turbine on to mechanical conveyors; it is then raised on to the sugar granary—an operation which is generally performed by elevator buckets (Fig. 74) similar to those employed in flour mills, or, when the sugar is in bags, by means of a crane. In the sugar granary the sugar is first dried, then mixed, screened, bagged and despatched.



*Drying the turbinéd sugar.*—Extra quality sugars are generally dried by spreading the sugar in thin layers on the floor, and systematically raking it about from time to time. Sometimes the sugar is run into a large revolving cylinder, through which a current of air is passed. When this is done the hot sugar from the turbine is run into it, and it comes out cold and dry.

*Method of mixing the sugars.*—The mixing of sugar was formerly done by laying it down in heaps, which were turned over by the shovel. In large factories this operation was done mechanically by

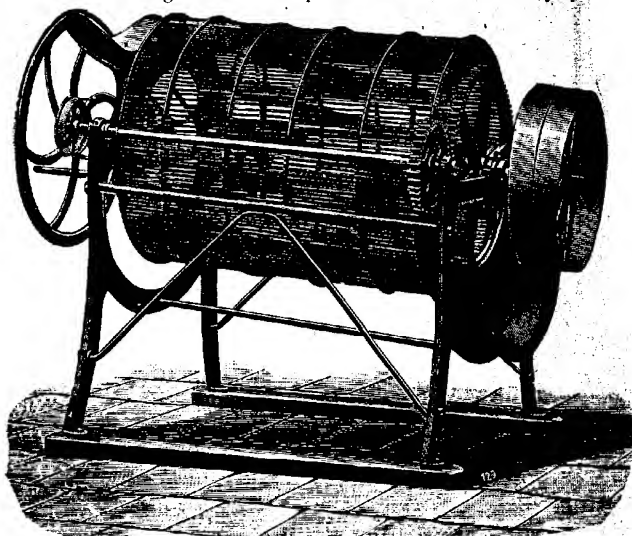


FIG. 73.—Sugar sifter (WATSON, LAIDLAW & Co.).

forming the heap on a trap-door on an elevated floor, which opened so as to precipitate the sugar on to the floor underneath, from which in its turn it is precipitated on to the next, so that all these successive falls sufficiently mix it.

*Screening and bagging up.*—The sugar was then screened, to keep out any core, then bagged, and in certain countries lead-sealed by the Excise, after which it was despatched.

But here again automatic methods have been brought into play, and manual labour reduced to a minimum. Fig. 74 speaks for itself.

*Denis's Automatic mixer of turbinized sugar (Fig. 74).<sup>4</sup>*—The object of this mixer is to render the various shades of turbinized sugar homogeneous, to reduce them to one uniform tint. Moreover, it embodies the most suitable, convenient and economical warehouse for storing

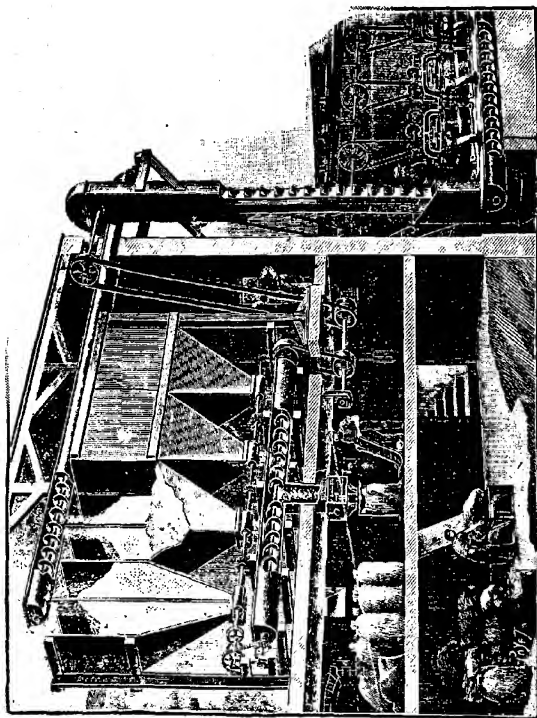


FIG. 74.—Denis's automatic conveyance of turbinized sugar to warehouse bins, core crushing rolls, helical mixer, and automatic bagging-up and weighing machine.

sugar before bagging-up. It not only breaks up the lumps of core, but regulates the feed of the automatic bagging-up and weighing machine. Floor-shovelling is suppressed, and with it all dirt, destruction and waste, as well as handling between the turbines and the bagging-up and weighing machine. The tight, rapid-measuring arrangement of this firm for charging turbines is well known, both

in sugar factories and refineries. It is shown in front of each turbine of the row illustrated in Fig. 74. The enamelled collectors, elevators and helices transfer the sugar to the warehouses in an absolutely inoffensive manner, without gumming, accumulation or breaking of the crystals. The enamelled helix and cups have none of the defects of rough screw conveyors, and under the propulsion of the enamelled spirals the sugar glides along as if in a porcelain tube, without accumulation or delay, so that an enamelled screw 10 inches in diameter does more work than a rough screw of 12 inches diameter. The following is the method of working : The sugar falls from the turbines into collectors and enamelled conveyors and ascends to the warehouse above by an enamelled elevator from which a helical conveyor carries it into the centre of the warehouse to be mixed and bagged up. Under this helix are the mixers. The arrival helix in the warehouse is fitted with trap-doors by which the sugar can be run into one or other of the six or eight square funnel-shaped coffers or bins placed under the helix and in one or two rows. The sugars of different shades are warehoused in these bins. A whole (*masse*) *cuite* may be stored in one bin or two distinct (*masses*) *cuites*. These warehouse coffers are fitted below with sliding doors through which when opened the sugar falls on to a helix under the door. This last helix runs the whole length of the six or eight bins comprising the mixer. It can thus collect the sugar falling from all the coffers. It collects apart those sugars which it is desired to mix by opening, say, the doors of the first and last bins, and so on. This mixing helix has inverse threads ; it brings the sugar from the extremities towards the centre, mixes the different shades, and brings them to the exit hopper above the automatic bagging-up and weighing machine. Turbined sugars from two, three or more bins may thus be mixed if desired. The regulation of the rate of discharge and the breaking up of the core are important points as well as the passage through the door of the automatic weighing and bagging-up machine. Denis gets over the inherent difficulties connected therewith as follows : Under each coffer is a pair of small wooden rolls propelled by a very simple motion. A chain connects one of the axes of the rolls with an axis of the adjacent helix. The two axes parallel and common to the two lines of the rolls are connected with each other by light gearing. The sugar which falls from the open bins drops on to the upper surface of these rolls, which are placed immediately below the sliding doors. Unless the rolls revolve, the sugar makes no motion towards the helix. The rolls must turn to impart the motion towards the mixing helix. The sugar now comes from the open bins, and falls in regulated amount on to the helix, which conveys it to the bagging-up machine. Any core present is crushed under each pair of rolls. To get good results in the breaking up of core and the regulation of the feed to the bagging-up machine, the inventor adjusts the rolls.

or better still, truncated rolls, so as to obtain a difference of speed between the reducing surfaces, and thus also to get more easily a greater or less distance between the rolls. This distance determines the quantity which in continuous working will allow 200 or 300 kilogrammes to pass down per minute. The conical form of the rolls facilitates this adjustment. The variation in the adjustment therefore allows more or less sugar to pass through. It is effected by sliding laterally—right or left—one of the shafts, not the driving one. At will, therefore, the space between the rolls may be varied from 5 to 12 mm. as demanded by the amount of sugar to be mixed and despatched. This sliding of the free shaft determining the shifting of the rolls is rendered practicable by using wider gearing to connect it with the first shaft, the driving one. By the same means reducing rolls, it regulates the quantity of sugar to be sent to the mixer, and afterwards to the bagging-up machine. One man suffices to attend to the whole work, fill, weigh and truck to the store 500 to 600 sacks in twenty-four hours. The Coche bagging-up machine, the concession of which is held by Denis, fills, bags up and weighs sacks to 100 kilogrammes, say 2 cwt., without the workman having anything to do but to hook on and hook off the bag and truck it to the pile whilst the next sack fills and regulates itself. This remarkable machine is original in construction and highly exact, and, verified by the French Department of Weights and Measures, it carries an index which shows in every case whether the weighing is exact. It is in use in over fifty of the numerous factories installed by Denis.

*Sugar warehouses.*—All the "firsts" sugars (see pp. 214-216) are mixed in the warehouses. The sampling and bagging up are done there. The bags are all weighed and lead-sealed by the Excise in certain countries. Generally this class of sugar is not stored in the warehouse; if it be not sold it is sent to the dépôt, where it is received, against a negotiable receipt, which facilitates trade without there being any necessity to touch the stored sugar. The "seconds" are sent to market in the same way; but the "thirds" remain a longer time in the store, owing to their tardy manufacture (p. 216). In those countries where only yellow sugar is made, even from the "firsts," sometimes the "firsts" and the "seconds" are mixed according to the percentage of sugar wanted. In Russia, where only extra-white sugars are sold, the "seconds" and the "thirds" remain in the warehouse, to be remelted next year, so as to get nothing but white sugar. The sugar warehouse is therefore the place where the commercial part of the factory is carried on, and should be capacious enough to contain a stock of manufactured products corresponding to the work of the factory, if the conditions of sale or storage in the dépôt do not appear satisfactory to the manufacturer, if the market price is too low, or the charges too high. These vary with the country, the harvests, and the temperament of the directors. There is no general rule upon the point.

## CHAPTER VII.

### EXTRACTION OF ALL AVAILABLE SUGAR FROM BEET-SUGAR MOLASSES.

*BEET-SUGAR molasses.*—*Their nature.*—The drainings from the turbinating of the last, *i.e.*, the lowest-quality sugars, constitute the molasses properly so called. These molasses still contain 50 per cent. of sugar. But the remainder of the substances in solution consist of organic and saline impurities, which prevent crystallisation of the sugar, and which are consequently termed *molasses-producing bodies* (*matières mélassigènes*).

*Disposal and packing.*—The molasses are collected in large reservoirs during the season, in vats, tanks or cisterns, run into casks (generally petroleum barrels), and sold to the distilleries.

*The sugar content.*—As in general 3 per cent. of molasses are obtained in beet-sugar factories, there is therefore  $1\frac{1}{2}$  per cent. of the original sugar of the juice still remaining in them (see detailed composition and method of analysing molasses, pp. 395 *et seq.*).

*Attempts to recover sugar.*—Different and sometimes economical methods have been tried to extract the sugar from the molasses, but the barbarous legislation which regulates the tax on sugar in some countries stifles any movement in this direction made by sugar manufacturers, under the pretext that they would thus produce sugar free of duty. The different processes proposed, adopted, then abandoned under the unjust taxes of fiscal systems, then resumed under more equitable legislation, have not become general, so that factories which extract sugar from molasses, or have an improved system in the working of which no molasses are made, are very rare.

*Abolition of molasses liberates vast working space now occupied by fill-houses.*—However, the abolition of molasses is a very important point in sugar factories, because it increases the yield, and does away at the same time not only with the long, tedious and clumsy work of the fill-houses, but with the fill-houses themselves, freeing in this way the vast spaces used for the crystallisation of low-quality products. The different processes proposed for this are legion. Those which have survived the test of actual working, and are met with in those privileged factories which have been able, either by means of

abundance of available capital or through the more enlightened intelligence of their owner, to transform their method of working so as to abolish molasses, specially claim attention.

*The old alcoholic method.*—If alcohol be mixed with molasses the major portion of the organic products dissolve, whilst the sugar, much more insoluble, is precipitated.

*The lime process.*—Another very old process consists in mixing the molasses with lime. A solid magma is formed, a kind of hard mortar formed by the combination of the lime with the sugar, under the form of an *insoluble tribasic sucrate*. By washing this magma with water or alcohol an insoluble sucrate of lime is obtained, and a liquid containing the greater portion of the impurities in the state of lime salts. In both these systems much sugar was lost in the wash-liquors. Those proposed since are only improvements—the practical perfecting of those old experiments with which the names of several celebrated chemists are associated, such as *Pesier*, *Marguerite*, and others.

*The osmogène.*—Finally, Dubrunfaut proposed and used a system based on the osmotic action of membranes (see pp. 35 *et seq.*), to separate the sugar in the molasses from the saline molasses-producing substances, which prevent crystallisation. The osmose process became famous, and reigned gloriously for a certain time; but the fiscal system killed it, and it has not been born again, in spite of the efforts of Leplay and the remarkable improvements he has brought to bear upon it. However, some factories still have their osmogènes, and still use them when the harvest, the price of sugar, or the fiscal difficulties permit, so that they must be regarded, as Horsin-Deon humorously points out, as amongst the living animals, like the dormouse, which wake up from time to time as fine weather comes round.

*The theory of osmosis or dialysis.*—When describing the diffusion process, the theory of the phenomenon termed osmose was explained—a phenomenon which induces the passage of two currents in a contrary direction through a membrane when its faces are in contact with solutions of different densities. This phenomenon, which occurs in plants through organised tissues, was tested by Graham on non-vegetable membranes or septa of animal origin, *e.g.*, bladders, in the apparatus termed endosmometers.

*Parchment paper as a membrane.*—Dubrunfaut applied the osmotic property to molasses by using parchment paper as a membrane. He noted the following results: If on one side of the membrane there be placed pure water and on the other side molasses, a *current of water passes to the molasses, whilst a saline current passes from the molasses to the water*. This saline current contains much salts, some organic matter, and a little sugar. In the beginning salts pass almost alone, later on the liquid contains a little sugar, still later the sugar

passes in greater abundance. If, therefore, the time of contact between the water on the one hand and the molasses on the other be limited, it is possible to extract from that molasses a large proportion of its salts and almost no sugar. Consequently the osmosed mass becomes crystallisable, and may thus be made to give up the sugar which the salts with which it was impregnated hitherto kept in solution.

*Description of Dubrunfaut's osmogène.*—It was to accomplish this that Dubrunfaut constructed his *osmogène*. It consists essentially of a series of parallel chambers in juxtaposition, having the form of a thin parallelogram, in which water circulates in the one and molasses in the other, the whole being kept hot, for *osmosis is much more rapid when the liquids are hot than when they are cold*.

The molasses chambers are intercalated between the water chambers, one between every two. The liquid enters below and flows upwards, following a zigzag movement induced by divisions made in the frames. In the parallel chamber the liquid follows the reverse direction. All the frames work separately, and thus form elementary *osmogènes* joined side by side into a single machine. By suitably regulating the molasses and the water-supply taps, the osmosis may be pushed to any desired point.

*As the water becomes charged with salts it increases in density. But the molasses diluted with water diminishes in density.*—The apparatus is regulated by taking the density of the two liquids at their exits, after having found by analysis the density at which to stop. Osmosed molasses and exosmosed water are thus obtained. To purify the molasses as much as possible, the osmosis must be pushed rather far. Therefore the exosmosed water contains much sugar. If it be concentrated and reosmosed, reosmosed molasses are obtained—which may again be crystallised—together with fresh exosmosed water which may again be osmosed repeatedly. Three or four successive osmoses are made in this way, and a very saline molasses is finally obtained which still contains sugar, but which is used more especially in the manufacture of potash salts. Most of the potash used in France in soft-soap manufacture is obtained from molasses. The wooden *osmogène* is cheap, and is of great service when it is known how to take advantage of all its capabilities. Unfortunately, legislation has banished it from France. Some are still in existence in Belgium and Russia.

*Leplay's improvements on the osmogène.*—Leplay, the worthy successor of Dubrunfaut, has made some improvements on the *osmogène*. The great defect in the *osmogène* is the change of density of the molasses, which enters at a high density and issues diluted with water, so that in the frames the density of the liquid is greater below than above. But as osmosis only goes on at a certain density, the purification is thus better at the bottom than at the

top. A portion of the surface of the frames is therefore, so to speak, lost.

*Evaporative osmogène.*—Leplay has therefore constructed osmogène-evaporators, which consist of ordinary osmogènes surmounted by a basin with a steam coil, in which the osmosed molasses continuously returns, and is brought back to its original density. This basin is formed by the frames themselves, which, pressed against each other, form on the top a plane surface with rims formed by the prolongation of the mountings of the frames. The evaporating osmogène has a considerable osmotic capacity, and yields remarkable products, both as regards richness and uniformity.

*Steffen's sucrate of lime separation process.*—The only process which has survived fiscal red-tapeism is that of Steffen, an Austrian. It is used in only a few factories, although it succeeds admirably with those who know how to work it. Its initial installation runs rather dear, but manufacturers who have enough capital to pay for this lucrative luxury find it to answer well. It consists in treating the molasses in the cold by powdered quicklime. When the degree of dilution is well adhered to and the temperature sufficiently low, below 15° C. (59°·2 F.), and the lime good, an *insoluble sucrate of lime* is formed which contains a little more lime than the *tribasic sucrate*. The impurities remain in the liquid. There are thus obtained (1) a liquid mud, containing sucrate of lime, which is filtered, pressed and washed in the press, and (2) a mother-liquor which contains all the salts, organic bodies, and a little dissolved sucrate of lime.

*Separation of the sugar from the sucrate of lime by carbonatation.*—There are two methods of separating the sugar from the sucrate of lime. One consists in mixing the insoluble sucrate with water and carbonatating it. This is not a good system, owing to the difficulty of effecting carbonatation of the insoluble sucrate.

*The solution process.*—In the other method, the solution process, the sucrate is dissolved in a sugar liquor. *Soluble monobasic sucrate* of lime is formed which dissolves in the liquid, and the excess of lime is precipitated. They are separated by the filter press, and the liquid carbonatated. But the solution system is not wrought by itself, but with the ordinary carbonatation of the juice.

*Dovetailing the recovering of sugar from molasses into the carbonatation process.*—Generally the molasses are wrought up during the period of treating the beets. Saccharate of lime is made in an annexe of the factory from the molasses, and used in both first and second carbonatations in place of lime. Both carbonatation and solution are effected simultaneously, the juice is enriched in sugar, and no special plant is required for treating the saccharate. If only so much molasses, as correspond to the lime required for liming the juice, be treated by the separation process, very good results are obtained.



*A portion only of the molasses can be advantageously used up in carbonatation.*—But sometimes the recovery of the sugar from the molasses is pushed too far. Again the saccharate still contains organic impurities, whatever care be taken in washing. These modify the composition of the juice, and the factory works badly. A check is thus courted by the production of impure syrups. It is far better, if there be an excess of sucrate left over, to dissolve it with the juice of the second carbonatation, and to pass it to the filter presses to separate the excess of lime, and then to carbonate those liquids. It gives more trouble, but it is safer. The best process is not to convert more molasses into saccharate than the safe treatment of the juice necessitates. (For chemistry of sucates, see pp. 73 *et seq.*)

*The details of Steffen's separation plant.*—The essential part of the separation plant consists of a *refrigerating mixer* (Fig. 75). This is a tubular body which exactly resembles a vertical triple-effect vessel, jacketed; cold water—as cold as possible—circulates in the jacket and in the intertubular space. It is surmounted by a hopper, from which it is separated by a revolving measurer, for introducing the powdered quicklime. A strong agitator keeps the liquid in motion.

*Grinding the lime underneath flat stones.*—A crusher near the lime kilns reduces the lime to coarse grains. This granulated lime is then ground underneath flat stones like those of a flour mill, then passed into sifting machines, from which it issues in the state of a very fine impalpable dust, which flies about in every direction. It is this lime flour which is introduced into the refrigerating mixer.

*Weighing the molasses.*—On the other hand, into a tank placed on a weighing machine the amount of molasses to be introduced into the mixer is carefully weighed, whilst the water required for dilution flows in until it reaches the desired level. There is thus about 3,000 litres of mixture.

*Measuring the powdered lime and feeding into the machine.*—*Mixing.*—When the mixer is full and at work the lime is added by means of the measurer. Each time that a measure of lime is introduced the temperature rises, and it is allowed to fall to 12° to 13° C. before the next addition of lime. When the density of the liquid shows that no more lime is required, the machine is allowed to make a few more turns and the operation is finished.

*Filter-pressing the mixed produce and converting it into sucrate milk.*—The contents of the mixer are then passed to the filter presses, washed, the cakes placed in another mixer, where they are mixed with water, and the resulting sucrate milk is used for carbonatation instead of milk of lime.

*Treating the mother-liquors.*—The mother-liquors from the filter presses still contain a little sugar. They are brought to the boil. *Tribasic sucrate* is formed, which is collected in the filter presses and added to the sucrate.

The above processes collectively form the separation process, the very simple working of which renders it very practicable. It, however, requires sustained attention and a continuous analysis of molasses. The works chemist is the mainstay of this operation, the process varying with the raw material used.

*The alcoholic method of extracting sugar from molasses.—Manoury's process.*—The alcoholic processes of extracting sugar from molasses, already referred to on p. 225, have nearly all been abandoned. There, however, remain a few applications of Manoury's system in Germany and Russia which require special mention.

*Treats the molasses with lime and elutriates with 40 per cent. alcohol.*—Manoury's process consists in preparing a solid combination of the molasses with lime in a granular form, and in treating the granular mass with alcohol in diffusers or elutriators. The special form of calcic compound formed with the molasses is obtained by adding to the molasses, contained in a horizontal mixer, powdered slaked lime obtained by dipping lime in lumps into water, and allowing it to slake without any further addition of water. The molasso-calcic granules thus obtained are placed in the elutriators. There are four of them, each containing 8 tons. Then alcohol is circulated so that the special diffusion lasts twenty-four hours. For 100 kilogrammes of molasses 29½ litres of 40 per cent. alcohol (say 29½ gallons per 100 lb.) are used. The alcohol, having done its work, is rectified. That remaining in the elutriators is driven

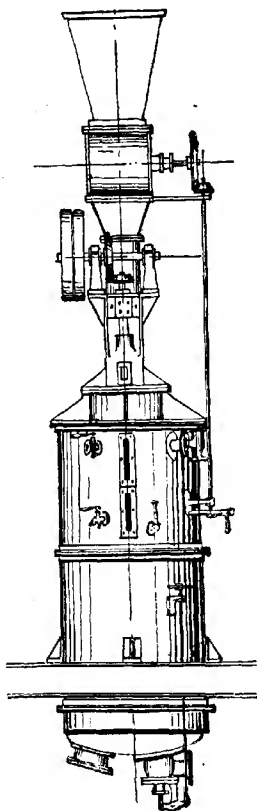


FIG. 75.—Refrigerating mixer used in the separation process (F. OLIN).

over by steam, and likewise collected. There then remains in the vessels a *sucrate* mud fit for use in carbonatation.

*Loss of alcohol in process.*—The loss of alcohol is the chief difficulty. However, Manoury has so well organised his plant that the leakage of 40 per cent. alcohol is only 2 per cent. Well-equipped factories which have erected this system have preserved it in spite of new methods invented too soon to have allowed the alcohol process to become to any extent general.

*The strontia method of extracting sugar from molasses.*—Another process which was very much in vogue, and which still exists in Germany, is the extraction of sugar from molasses by strontia. Though the *Raffinerie Parisienne* still works by this process, strontia is too dear for it to become general.

*Advantages of sucrate of strontia over sucrate of lime.*—Strontia sucrate is much more easily formed than lime sucrate. That is why the celebrated German chemist Scheibler pressed its use in sugar factories, the more so as Germany has extensive strontia deposits, where it can therefore be obtained very cheaply.

*Description of the strontia method.*—The operation is very simple. In a vessel, cooled to the surrounding temperature, the molasses are mixed in suitable proportions with the strontia. Sucrate of strontia is formed, the mother-liquors are separated by filter presses, the cakes washed in the press with cold water, and sucrate of strontia cakes are thus formed, which are afterwards mixed with water and carbonated. The carbonate of strontia is returned to the caustic condition and used over again. All this can be very rapidly described, but the application of this principle is tedious. The mother-liquors have to be worked up, for they still contain strontia and sugar. The reurning of the strontia is not convenient; the operation as a whole requires extensive plant, because it is necessary to make the strontia and to reurn it to save expense.

*Tends to be superseded by Steffen's process.*—However that may be, the strontia process has its advocates, although much less is heard of it since the introduction of Steffen's process, which presents many advantages over it.

*Influence of continuous sulphitation of the syrups and drainage liquors on the viscosity of molasses.*—The practice in sugar factories of returning the drainage liquors to the "firsts" vacuum pans and the different processes leading to the production of molasses in manufacture have demonstrated the necessity of eliminating as far as possible the viscosity of the "firsts" and "seconds" syrups. The most general method is to treat them with sulphurous acid. The Quarez system (Fig. 76) presents numerous advantages: (1) The sulphurous acid is drawn into the syrups by aspiration, and not forced into them by compression. (2) The quantity of syrup in contact with the sulphurous acid is very small, 3 to 4 hectolitres

only can undergo a false manipulation. (3) The entrance and exit of the syrups being continuous, the sulphitation may be stopped or started instantaneously; the work of the factory can never be stopped thereby, the syrups passing through the apparatus whether sulphited or not. (4) The apparatus may be very easily installed, occupies but little space, and lends itself to every combination of working. (5) Any workman, even a child, can work the machine.

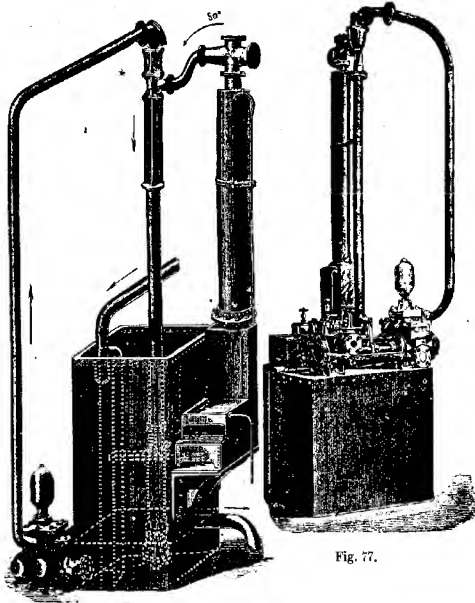


Fig. 76.

Fig. 77.

FIGS. 76, 77.—Quarez's system of continuous sulphitation (MAQUIN).

(6) It has no valve in contact with sulphurous acid. Fig. 77 shows the apparatus used for the syrups of a beet-sugar factory working up to 300 tons a day, the furnace and the pump on the sulphitation tank itself. The necessary space is reduced to 4 feet by 4 feet. Fig. 76 shows the apparatus employed for drainage liquors, and in the case of factories producing more than 300 tons. The trunk is replaced by a special injector of great capacity, and the tank is higher

to allow for the expansion of the froth which is produced so abundantly in the sulphitation of the drainage liquors. The furnace and the pump are then placed either on the floor or at a sufficient height. The apparatus is simply regulated by the speed of the pump, which is increased or decreased according as the sulphite workman finds it more or less alkaline to litmus paper. The quantity of sulphur burnt is in proportion to the draught induced by the passage of the syrups into the injector, and consequently the speed of the pump. According to the output of the factory the apparatus is made with two, three, four, or more injectors, and even in this case the necessary space is reduced to a minimum, and nothing in comparison with the encumbrances incidental to other processes.

During the season 1899-1900 sulphurous acid was used in no less than 162 sugar-beet factories in France in the *purification and defecation of the juice*. For use of sulphurous acid in purification of cane juice see p. 278, and in sugar refining see p. 297.

*The elimination or avoidance of molasses-making in beet-sugar manufacture.—Crystallisation in motion.*—On the principle that prevention is better than cure, rather than try to extract sugar from molasses it seems natural to strive not to so degenerate the sugar as to produce molasses. As molasses is the result of the non-crystallisation of the sugar under the so-called molasses-producing influence of the organic and saline impurities in the juice, the problem therefore is how to eliminate these impurities. Carbonatation eliminates the greatest possible bulk of the organic matter and some saline impurities, but the bulk of the salts remain behind in the juice. Dubrunfaut proposed the previous *osmosis* of the juice to eliminate from it the bulk of the saline matter, but this osmosis, however little, *lost too much sugar*. Up to now, therefore, it has not been found possible to eliminate all the organic matter, far less the salts. If it be not practicable not to make any more molasses, *it is at any rate necessary to find means for making the sugar crystallise in the midst of its enemies, the molasses-producing bodies*. Then molasses will still be produced, but in such trifling quantity as not to be worth mentioning.

Two methods have lately been tried to reach this end. One, *crystallisation in motion* (Figs. 58-60), permits this principle to be applied in all factories without changing the usual routine—a few more engines, that is all. The other—*Steffen's new process*—changes the whole of the final part of the work, starting from the boiling, and involves extensive and dear plant. But it has the advantage of making all the extractable sugar white, whilst the first method makes as many grades of sugar as it does vacuum pan strikes. When a saccharine solution is left to itself at a temperature of 30° to 40° C., and sufficiently concentrated, say about 40° B. (sp. gr. 1.376), this syrup crystallises slowly. Each crystal formed is fed at the expense of the surrounding solution, which gets weaker and weaker, until

the solution is too weak, and abandons no more sugar. The sugar and foreign matters are then in the ratio of 3 or 4 to 1. Dubrunfaut said that one part of saline matter prevented 3.75 of sugar from crystallising. *Feeding the crystals.*—Some years back it was observed that in stirring syrup, in presence of sugar crystals in the form of a bait or nucleus, and cooling the syrup, the affinity between the sugar and the molasses-producing agents was greatly reduced, and that the yield in crystals was therefore greater. Every one knows, in fact, that when sugar syrup is stirred with a spatula the syrup rapidly crystallises into a compact mass. This was, in fact, the only way the natives used to crystallise their crudely made and roughly evaporated cane juice. Taking advantage of this fact, a new process of crystallisation in beet-sugar factories has quickly been established, for quickly cooling the "firsts" *masse cuite* before turbinising in the *masse cuite* receivers for crystallisation in motion—already described (pp. 186-190). It was adopted almost simultaneously in Germany and Belgium (see also Figs. 58-60 and context).

*Reciprocal crystallising effect of the masse cuite syrup and the feeding syrup.*—Working upon the fact that syrup crystallises quicker and exhausts itself of sugar to a greater extent when agitated, poorer syrups were poured on to the "firsts" *masse cuite* and stirred by an agitator; the poorer syrups gave up their sugar to the crystals already formed, so that both the syrup of the boiled mass and the added syrup were simultaneously exhausted of sugar. Therefore, if this new mass be turbinised a much greater yield of white "firsts" is obtained than in the usual way of working, and to make additional "firsts" is a step in advance. The molasses coming from the turbinising of the "firsts" is added to the next *masse cuite*. So that, if the process went on indefinitely, the final result would be "firsts" and molasses. But this syrup very soon becomes too impure. The molasses is then liquefied, either all at once or successively, and boiled as "seconds," which is crystallised in the same way. Finally, very poor "thirds" is obtained and very poor molasses. *Crystallisation in motion* is being more generally adopted. Figs. 59 and 60 show *masse cuite* receivers for crystallisation in motion, whilst Fig. 58 shows general arrangement of cane-sugar plant for the working of the crystallisation in motion process.

*Steffen's systematic process for reducing molasses production to a minimum.*—This process was in use in France in three factories in 1899-1900. It was invented by an Austrian, a very keen observer of great merit, named Steffen. The process consists of a system of boiling in grain, and treating the boiled mass so as to convert all the sugar into "firsts" sugar, and only to make but the minimum quantity possible of molasses.

*The process entails the alteration of the whole treatment of the juice after it comes from the triple-effect.*—From the stage where the syrup

issues from the triple-effect vessel the ordinary method of manufacture is altered.

*Feeding the boiled mass with fresh rich syrup.*—Steffen observed that the first crystals formed in the vacuum pan are produced in the midst of a liquid, in which the ratio of the sugar to the non-sugar is considerable. But as soon as the crystals are formed the medium in which they swim becomes poorer and poorer in sugar. To feed the *masse cuite* with fresh rich syrup is to change the mode of existence of the crystal, to induce the birth of new crystals, which one is obliged to melt again in reheating the mass; to bring about in fact a perturbation in that which is already done. Rationally, it would be better to feed the *masse cuite* with poorer and poorer syrups of the same purity as the syrup existing in presence of the sugar at the moment of feeding. This is what Steffen does in his methodical boiling. But to get these results it is necessary to have syrups of graduated richness. The method of obtaining these will be given in the sequel. If the mass be fed with poorer and poorer syrups the last syrup will be nothing but molasses. If this mass be turbinéd, nothing is obtained but white sugar and poor molasses, which need not be reboiled as it would yield no more sugar. By a single operation, therefore, in the vacuum pan it has been found possible to make white sugar and molasses, suppressing the working of the poorer products, fill-houses, etc. The manufacture finished, the factory may be closed. There is nothing further to be done until next season.

*Supersedes turbines by methodical washing.*—To obtain his graded syrups, Steffen dispenses with turbines, by a methodical washing of the *masse cuite* in special vessels with cleansing liquids of a greater and greater degree of purity, which mutually follow each other, until only pure sugar syrup is used as the last cleansing agent. The *masse cuite* cleansed in this way, drained and dried, is nothing less than very pure white sugar. As these successive cleansing liquids get more and more bulky, the excess is used to feed the vacuum pan.

*The molasses removed by exhaust from wire-gauze-jacketed tanks.*—The process is conducted in the following way: The *masse cuite* is run into a series of jacketed flat tanks on wheels; the jacket consists of wire gauze analogous to that used in the turbines. First of all, an exhaust forces the molasses which soils the crystals to rapidly pass through the jacket. This molasses is the final product, and is sold to the distillers.

*Further cleansing with sugar syrup.*—When the molasses has been eliminated a syrup richer in sugar is poured on the mass, then a richer still, and so on to a pure sugar syrup.

*The methodical using up of the spent sugar syrup cleansing fluids.*—The second cleansing syrup passed is weakened by being mixed with

the molasses remaining between the crystals; it may be used as the first cleansing liquor of the following operation. The third cleansing liquor becomes the second, and finally the sugar solution will become the second last.

*The storage of the various cleansing liquors.*—All these successive cleansing liquors are stored in a large tank, with compartments fitted so that the different liquors flow simultaneously on all the waggons at one operation; all that has now to be done is to empty the waggons, which no longer contain anything but white sugar. This emptying is done by tilting them upside down into a hopper, where a mixer imparts a little more homogeneity to the sugar.

*Turbining and drying.*—From the mixer the sugar falls into the turbines, where a cleansing by dry steam (Fig. 71) dries the crystals; if this be not sufficient, it is run into a large horizontal cylinder revolving on its axis, through which a current of warm air from an air propellor terminates the drying.

*Instalment expensive, but gives marvellous results.*—Steffen's process works very regularly and promises well. But it is very dear to instal. The results are none the less marvellous. It has therefore been adopted in some factories, in spite of the heavy disbursement for plant and royalty for use of patent.

*Combined with the inventor's separation process is still more costly and difficult to redeem.*—To complete this system and to win from it every possible advantage, it must be supplemented by Steffen's separation process. Because as now described it yields finally as much molasses as the ordinary process with simple turbinizing. However, by working the two processes conjointly, an extraordinary yield of sugar is obtained, all of which is white, with an insignificant residue of molasses. But by the addition of the separation process the method becomes still more costly, and it is a long time before such a large amount of capital is redeemed. Hence the delay which manufacturers exhibit in adopting these new inventions, whatever might be the profit they would gain by getting all their sugar as white sugar, and by being able to stop work the day the last beetroot is passed, and by abolishing the fill-houses and the expensive work incidental to the "firsts" and "seconds". But it is a great point gained, and no small consolation, to know that, the day the price of the necessary plant and machinery will permit of it, all beet-sugar factories will be able to work much more rationally than they do now; and, moreover, when the question has been better examined, and thus becomes better understood, means may be found to simplify all these operations and to reduce the cost of this expensive plant whilst arriving at the same result in the end.



## SECTION II.

### CANE SUGAR.

#### CHAPTER VIII.

##### THE SUGAR CANE AND ITS CULTIVATION—EXTRACTING THE JUICE BY MILLING.

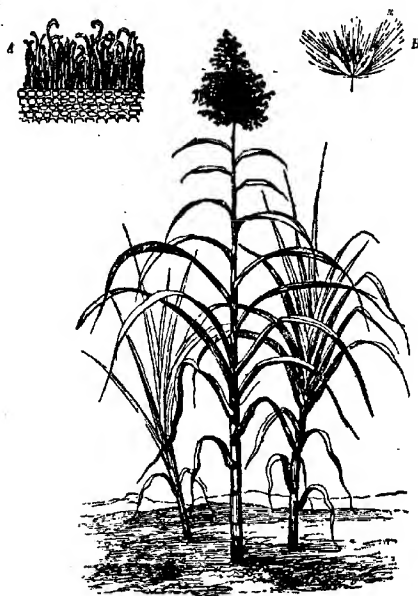
*THE sugar cane and its average richness in sugar.*—The sugar cane is a large-sized plant of the order *graminaceæ*, but differentiated from other *gramineæ* by the nature of its juice, which, in a suitable soil and climate adapted to its perfect growth and development, may contain as much as 18 to 20 per cent. of sugar. However, under less favourable conditions, as in Louisiana, U.S.A., the sugar in the juice does not amount to more than 15 per cent.—much the same as in the sugar beet. The structure or texture of the cane is graminaceous, the envelope coriaceous; the interior portion consists of vessels not very close to each other, but arranged to form a spongy mass, in the midst of which the saccharine saps circulate; nodes at the axils of the leaves, exceedingly hard at the base; a crown of foliage bearing a flower as the plant approaches maturity. Each node is furnished with an "eye" capable of reproducing a new plant when it is planted in the ground. That is the way the species is propagated or reproduced, because the cane under ordinary conditions does not bear fruit. It is therefore impracticable to reproduce it from seed. *The wild cane versus the cultivated sugar cane.*—The sugar cane is a *cultivated*, not a *wild*, plant. The juice of the *wild* cane, altogether a much smaller plant, is much poorer in sugar. The wild cane reproduces itself by seeds. Attempts, still in the experimental stage, have lately been made to regenerate the sugar cane by sowing wild species.

*Composition.*<sup>1</sup>—*Woody fibre cellulose and organic acids.*—The sugar

<sup>1</sup> The composition of beet juice is given on p. 71 and it may be useful to compare the two.

cane contains not only sucrose, but numerous other substances dissolved in water, as well as cellulose or woody fibre. The woody fibre contains about 70 per cent. pure cellulose ( $C_6H_{10}O_5$ ) and 30 per cent. wood gum or xylan. The following organic acids are

also present in the juice—glycollic acid,  $\begin{array}{c} CH_2-OH \\ | \\ CO-OH \end{array}$ ; malic acid,



(A—En crustation of wax on epidermis, magnified. B—Floret.)

FIG. 78.—The sugar cane.

$CH-OH-CO-OH$   
 $\begin{array}{c} | \\ CH_2-CO-OH \end{array}$ ; succinic acid ( $CO_2H-CH_2$ )<sub>2</sub>; and small quantities of tannic acid,  $C_{14}H_{10}O_2$ . Again, citric acid,  $C_6H_8O_7$ ; tartaric acid,  $C_4H_6O_6$ ; and aconitic acid,  $C_3H_3(CO_2H)_3$ , are said to be present in the normal sugar cane, but their presence has not been confirmed. Some authorities say the lime salts of these organic acids are soluble, and are therefore not removed by clarification, which seems rather impossible. The action of lime on the cane juice and its impurities

is highly complex; it apparently gives rise to the formation of lactic acid,  $C_6H_{10}O_5$ ; saccharinic acid, gluconic acid,  $C_6H_8(OH)_5CO_2H$ ; saccharic acid,  $CO\cdot OH - (CH\cdot OH)_4 - CO\cdot OH$ . As will be readily understood, acetic acid is only present in fermented cane.

*Albumenoid and other nitrogenous substances.*—The nitrogenous bodies in the cane are mainly albumenoids, amides or amino acids, and xanthine bases. It is uncertain whether the amides or amino acids consist of asparagine, aspartic acid or glycol. The xanthine bases consist chiefly of guanine ( $C_5H_5N_5O$ ), but the total nitrogen in the ripe sugar cane does not amount to more than 0.1 per cent.

*Colouring principles.*—These consist principally of chlorophyll and its derivatives and anthocyan, and a colouring principle which becomes yellow in contact with alkaline liquids.

*Cane-sugar wax or cerosin.*—The cane wax or cerosin (Fig. 78*d*) on the outside of the cane is said to be soluble in alcohol, ether, chloroform and benzine, but according to Beauvisage it is insoluble in both cold alcohol and cold ether, slightly soluble in boiling ether, but very soluble in boiling alcohol. It is got from the cane by scraping its stems, or, better, by purifying the skimmings of the non-limed juice. It is hard and brittle, with a clean fracture, and may be easily pulverised in a glass or marble mortar. Moulded into a candle, it burns with a fine white flame. It melts at  $82^\circ C$ . and solidifies at  $80^\circ C$ . It may then be crystallised in interlaced truncated needles. Its density is 0.961. On cooling its alcoholic solution, it may be obtained in fine pearly lamellæ; but this alcoholic solution becomes converted into an opaline mass on cooling; 20 centigrammes of cerosin are sufficient to solidify 30 grammes of alcohol and to give it the appearance of opodeldoc. The chemical composition of sugar-cane wax is but little known. Only its elementary composition has been determined, and a formula given to it corresponding to an alcohol of the fatty series or an ether. But there is nothing to prove that it is a definite compound and not a mixture of different substances, even although a recent writer describes it as a saturated alcohol with 24 carbon atoms.

*Valuation of sugar cane.*—1. *Sampling.*—The preparation of a fair average sample of the bulk of a lot of whole canes is difficult. Not only does the composition of different canes vary, but even that of the different internodes in one and same plant. A fairly large number of canes, representative of the bulk, must be taken, cut into rounds by means of a cane cutter, and from the cut bulk a fresh average sample taken and crushed in a mortar to obtain a more or less coarse pulp.

2. *Direct estimation of the sugar.*—The coarse pulp from 1 is not fine enough to admit of sucrose being estimated by the cold digestion process (p. 17). The hot process (p. 17) must be adopted, taking twice the normal weight for 200 c.c., and so on for 400 c.c.

according to saccharimeter used. Add 10 per cent. of weight of cane of basic acetate of lead solution in c.c. Fill flask to mark with hot water. Digest for an hour at least in an almost boiling water bath. It is best to fix a circular piece of metal, perforated in several places to prevent pulp rising out of the water. Cool. Polarise in a 400-millimetre tube. The correction for space or volume occupied by marc is about 1 c.c. for 10 grammes of canes, say 3 c.c. for twice 16.29 grammes, and 5 c.c. for twice 26.048 grammes.

3. *Exhaustion of the cane pulp by repeated washing.*—Weigh out 100 grammes of cane pulp, add 200 c.c. of water and a little carbonate of lime, and bring to boil. Draw off solution, add 150 c.c. of fresh water, heat again, repeat extraction during ten minutes, making six extractions in all, collecting liquid in a litre flask; add 8 c.c. of basic acetate of lead, cool solution, make up to mark, filter and polarise in a large tube. Calculate result to per cent.

4. *Indirect analysis.*—The process for estimating sucrose in cane juice is identical with that used for beets (pp. 12 *et seq.*). The cane contains about 10 per cent. of insoluble. The first pressing, *premier jus* or *vesou*, is the richest in sugar. To calculate the richness of the cane in sucrose from that of the juice, it is necessary to multiply by a coefficient varying between 0.87 and 0.88. To ascertain the industrial richness of the cane, apply the coefficient 0.85 to the sucrose in the juice to allow for various losses and waste which the cane suffers from the time it is weighed until it comes to be treated.

*Culture of sugar cane.*—*Reproduction not from seed but by buds.*—The sugar cane is not reproduced like ordinary graminaceous plants, but is reproduced by buds. When the cane is ripe it is cut down level with the ground. But, before carrying it to the mill, the crown (Fig. 78), with its dense foliage, is cut off; and the nodes remaining on this part of the stem are generally used as buds for the propagation of the plant—a bad practice, conducive to the degeneracy of the species. The ripe portions of the cane produce much better shoots than the nodes of the badly developed unripe crown. It is the adoption of an imperfectly understood false-economy policy.

*Each plantation should have its propagation field or nursery.*—It would be far better to set a field apart for propagation purposes than to use portions of the cane too weak to sustain the exhausting efforts of a healthy and vigorous reproduction.

*Planting the buds.*—The bud is planted in the ground in the bottom of parallel furrows of 6 to 8 inches in depth, in moist weather, so that the germ may shoot up rapidly. They are not placed horizontally or flat in the ground, but by raising one of the ends to about 45°. They are then covered with loose soil.

*Fertile soil and a fine state of tilth absolutely necessary.*—The cane requires fertile, well-manured ground, but planters often neglect the

most elementary principles of agriculture; and the fields, in bad cultivation and preparation, and without any manure whatever, only yield but very indifferent crops. The cane requires the same state of tilth a crop of wheat requires, and the crop is proportional to the care and labour bestowed upon it.

*Irrigation as an aid to successful sugar-cane cultivation.*—In warm climates with little or no rainfall the cane cannot sprout and shoot up without the aid of systematic irrigation. In Egypt sugar can only be cultivated in certain districts by aid of irrigation canals, fed continuously with the water of the Nile from artificial dams.

*After cultivation.*—After the cane is planted it soon begins to shoot forth a tuft of canes, and, after several hoeings, the fields are

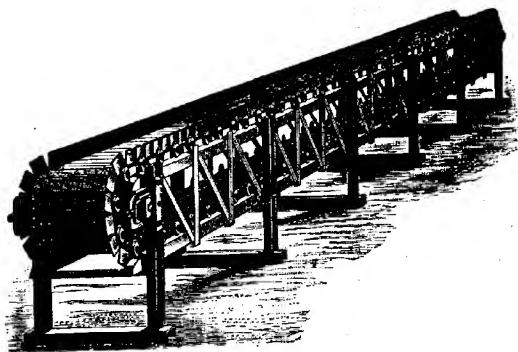


FIG. 79.—Cane conveyor (CAMBRAY).

covered as with a dense forest, each stem being 6 to 15 feet high, and from  $\frac{1}{2}$  to 2 inches in diameter.

*The sugar-cane harvest.*—When fully developed the sugar cane is cut down by strong knives level with the ground. The crown, with the nodes immediately underneath it, are removed, because that part, like the neck of the beet, is more impure than the remainder. The leaves are stripped and left on the ground. The canes thus prepared are transported to the factory by the conveyor (Fig. 79), or by Bullivant's cable system, or in railway waggons drawn by a locomotive. Being very bulky and cumbersome, it necessitates a large space in connection with the sugar factory in which to store it, but in any case it should be treated as soon as possible after its arrival in the factory yard.

*The stubbles.*—After the cane is cut, the stubbles remain in the

ground. But they are still alive, and three years afterwards may still yield an abundant harvest. The fresh plants are thus cropped for three years or even longer. The planter should therefore devote every care and attention to the fundamental portion of the work, without which good returns cannot be secured. But if this initial preparation be well conducted the planter is guaranteed a long series of profitable crops. The discriminate selection of the plant is therefore necessarily a fundamental step of vital importance.

*Cost of sugar-cane cultivation and milling in Cuba.*—Good sugar-cane land may (1902) be bought in every province of the island, but best results are to be got in virgin soil in Santa Clara, Puerto Principe and Santiago. As cane has to be cultivated on a large scale, and as the unit "caballeria" ( $33\frac{1}{3}$  acres) is convenient when calculating on large tracts, it will be here used as a unit of measurement. The cost of cultivating a caballeria should be about as follows:—

Clearing and ploughing . . .	\$500, say = £100
Seed cane purchased . . .	80 " 16
Planting . . .	200 " 40
Cultivation . . .	300 " 60
	<hr/>
	\$1,080 " £216

$33\frac{1}{3}$  acres therefore cost £216, therefore 100 acres cost £648, or say £6 10s. per acre, or about three times as much as it costs in Great Britain to raise a crop of wheat. If timbered land be selected and cleared, the woods therefrom should yield at least \$600, say £120 per caballeria, that is, £360 per 100 acres, or £3 12s. per acre; about half that amount should be the initial purchase price. Consequently the end of the first year should show the land itself as a clear balance to the credit of the planter, with a growing crop of cane representing the capital invested. Every caballeria of virgin land should yield 1,200 tons of cane to cut and haul, which would cost about \$1 per ton. This amount of cane should produce at least 120 long tons of sugar, or 268,800 pounds. A liberal estimate for milling is  $\frac{1}{2}$  cent per pound; the actual cost on a modern plantation is nearer one-fifth, or \$1,344 for producing this amount of sugar. The balance sheet at the end of the first harvesting should show for every caballeria:—

Dr.	Cr.
To making and harvesting crop \$2,280	Sale of 120 tons sugar @ \$44.8
" milling 1,200 tons cane . 1,344	per ton . . . . . \$5,376
Total cost . . . . . 8,624	
To profit . . . . . 1,752	
	<hr/>
	\$5,376

\$1,752 is, say £350, or £1,050 per 100 acres, say £10 16s. per acre. After the first crop is milled the total annual expense will be only about \$1,500 per caballeria, thus increasing the profit to \$2,532, say £500, or £1,500 per 100 acres, or £15 per acre. The authority who gives these figures says this is unquestionably a very attractive showing, but is based on thorough knowledge of sugar-cane culture and sugar manufacture, and careful comparison of data furnished by men actually engaged in the industry in Cuba. There is risk of loss by fire or storm, although cane is considered about the safest from such injury of all crops produced in Cuba. But the individual or syndicate undertaking its cultivation and conversion into sugar must remember that much capital has to be invested in expensive plant and buildings, that the crop itself needs careful attention from planting to harvesting, and that details of management should be entrusted only to experienced competent men.

TABLE XLV.—THE YIELD OF SUGARS PER TON OF CANE IN LOUISIANA, U.S.A.

Description.	Yield.	Sugar per Ton of Cane.	Yield.	Sugar per Ton of Cane.
	A.	A.	B.	B.
	lb.	lb.	lb.	lb.
First sugar (white) . . .	116,851	88.22	163,539	87.46
Second sugar (yellow C) . .	63,008	47.57	88,814	47.49
Second sugar (waggon) . . .	...	...	6,833	3.12
Third sugar (waggon) . . .	26,601	20.08	84,295	18.84
Fourth sugar (waggon) . . .	15,126	11.42	21,355	11.42
Total . . .	221,586	167.29	314,836	167.84

A, first run, November 12 to 18 inclusive: cane worked, 1324.5 tons; mill work, 1217 tons; diffusion work, 107.5 tons. B, second run, November 19 to 30 inclusive: cane worked, 1870 tons; mill work, 1456 tons; diffusion work, 414 tons.

*Milling results in Louisiana, TABLE XLV.*—The character of the season (1888-89) producing exceedingly woody cane and a small tonnage accounts for juices being so rich that season. The first few analyses showed a high percentage of glucose, due to deterioration of cane left on yard during preliminary work. Delays were unusually frequent at beginning of season, hence considerable cane was left on the yard for several days. The percentage of available sucrose, based upon an extraction of 72 per cent., and calculated by the formula "per cent. sucrose minus one and one-half times the glucose

= available sucrose," was 10.31. The available sucrose in lb. per ton of cane =  $206.2 =$  approximately 214 lb. of commercial sugar. The actual yield of sugar obtained was about 45 lb. per ton of cane less than this. Not having reliable data of the mill work, it is impossible to locate responsibility for this shortage. Possibly it was partly due to a lower extraction than 72 per cent., but this alone would not account for the shortage.

Very likely the class of sugars made also accounts in part for low yield. The "firsts" generally graded as "choice white". To obtain this grade, it was necessary to use much water in the centrifugals. The "seconds" were graded in the pan. The "firsts" molasses were diluted, treated with superphosphate of lime and alumina, relimed and filtered through bone black. Again, con-

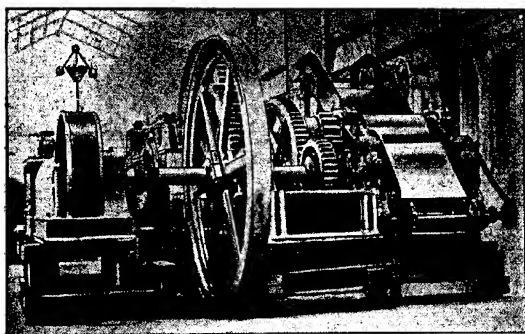


FIG. 80.—Sugar-cane mills (CAIL).

siderable water had to be used in the centrifugals to obtain a high grade of sugar.

The sugar cane may be deprived of its juice by two essentially different methods:—

1. *Milling*.—The first method is by the ancient milling process, i.e., by expression in a mill, consisting of a set of powerful rolls which crush the cane in its passage through them, and the liquid juice is squeezed out at the same time by the strong pressure which even crushes the very hard nodes of the cane. 2. *Diffusion*.—The second process by which the sugar cane is made to disgorge its juice is the essentially modern one of diffusion. (1) *Sugar-cane milling*.—But it will be desirable to treat of the milling process in the first instance. In olden times cane mills consisted of two vertical wooden



rollers driven by a horse, the cane being passed between them. Modern engineering has replaced this primitive appliance by strong mills driven by steam power, the rolls of which extract a far larger quantity of juice than the rudimentary mills of former times. Moreover, the mills have been supplemented by an arrangement by which even still less sugar is left in the cane. The centre of the cane is spongy, and every one knows that when a sponge is pressed, the sponge again drinks up a portion of the liquid with which it was



FIG. 81.—Milling followed by immersion. Vertical section (CAIL).

prepared to part if the pressure had been a little more strong. The cane was therefore made to imbibe water and pressed a second time, yielding a fresh saccharine liquid, more dilute, but none the less profitable.

*Cane shredders.*—Machines have been invented, called shredders, which tear the cane up into fibres more easily moistened and pressed, and yielding a still larger quantity of juice. As much as 90 per cent. of the juice in the cane may thus be extracted, whilst the mill itself can only express some 70 to 75 per cent.

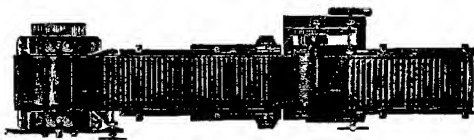


FIG. 82.—Milling followed by immersion and subsequent remilling. Plan (CAIL).

*Description of a modern horizontal sugar-cane mill.*—A sugar mill consists of three horizontal cast-iron rolls arranged thus: The axes of two of the rolls (A and B, Fig. 84) are in the same horizontal plane, and are quite near to each other without touching, yet are sufficiently far apart to leave between them a space, varying with the size of the mill, between 8 and 10 inches. The third cylinder (C) is superimposed on the two others so as to touch them both. The axes of the two cylinders (Figs. 82 and 84) are held in position.

at their extremities in stuffing boxes in a very strong cast-iron frame. The distance between the rolls may be varied by pressure screws. Strong pinions render the three rolls firm and solid so that they turn together, acting as one only—the top one. Gearing and

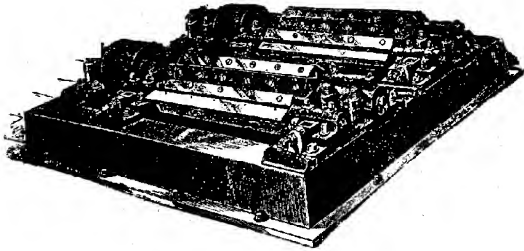


FIG. 83.—Cane shredder (CAMBRAY).

shafting (Fig. 80) connected with the axis of the top roll are driven by a steam engine, so as to impart the right speed to the rolls. The cane is brought, by an inclined plane (Fig. 84, D), between the top roll and one of the bottom ones. The rotation motion brings it under the top roll, and from there it passes between the same roll

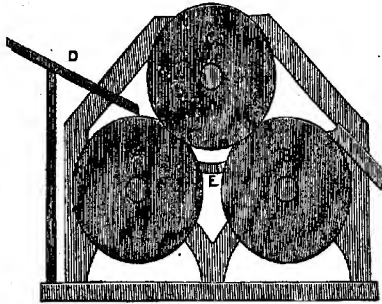


FIG. 84.—Diagrammatic representation of cane mill.

and the second lower roll, being led in that direction by a guide plate E placed between the two lower rolls. Finally, it makes its exit, and falls on an inclined table, and afterwards on a conveyor which carries it outside the factory. To carry the cane to the mill,

a long mechanical conveyor (Fig. 79) rolls in front of the work-people, who throw the canes into it by armfuls, and it thus arrives in front of the inclined plane (Fig. 84) which feeds the mill, or it may be brought by locomotive on waggons running on a light railway. The cane conveyor and the bagasse conveyor are driven by the mill itself so that, when it stops, the feed also stops.

*Size of mills.*—The sizes of mills are very variable indeed. Some are very small. But ordinary mills have rolls 30 inches in diameter and 78 inches long, crushing 250 to 300 tons of cane in twenty-four hours. The rolls make two revolutions a minute. But in the Rodah factory, Egypt, close on 2,000 tons are milled daily, and even larger quantities in Cuba, Honolulu and Demerara.

A diagrammatic representation of the mechanical principles of the three-roll horizontal cane mill is illustrated in Fig. 84. A, B, C are three cast-iron rolls with flanges at their ends to prevent the escape of the canes. In the case of large factories these rollers are very

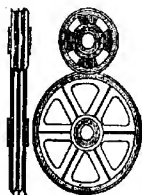


FIG. 85.—Friction wheels.

massive and ponderous, attaining the dimensions 6 feet in length by  $2\frac{1}{2}$  feet in diameter. The upper roller C is occasionally fluted longitudinally so as to get a good firm grip of the cane, and thus make sure of drawing it into the mill. But these longitudinal grooves are not indispensable. As the grooves hash up and crush the cane very roughly and otherwise injure it, the smooth uniform surfaced roll is preferred. The three rolls revolve simultaneously, being driven by means of cog-wheel gearing.

Either steam, wind, water or animal power may be used. D is an inclined table down which the canes descend to reach the first set of rolls A, C. These rolls are about quarter of an inch apart. The distance may be regulated by screws. The cane is well crushed between the first set of rolls A and C, and converted into a fit condition to benefit by the extreme pressure which is afterwards imparted to it in its passage between the rolls B and C. At E is a guide plate upon which the crushed canes are deflected upwards, after passing the first set of rolls, so as to cause them to pass between the rolls C and B. These rolls are much closer together than A and C—so close, in fact, that a sheet of paper can hardly be pressed between the two. Under this energetic pressure the juice is squeezed or expressed from the cane, and drips from the rolls into a gutter, which conveys it to vessels where its further treatment is begun. The rolls lying so near to each other will be more or less liable to get jammed when irregularly fed with too bulky a charge of cane. A sudden stoppage of this kind would lead to a serious break-down—either the rolls themselves would be

injured, or the cog-wheel gearing or shafting by which they are driven. Various measures are adopted to avoid this calamity. It has been even suggested by Sir Robert Ball to drive the rolls by friction wheels, the nature of which will be readily perceived from the accompanying illustration (see Fig. 85). These, instead of being on the tooth or cog-wheel principle, have a series of ridges on their circumference which fit into a corresponding series of ridges. When a pair of wheels constructed in this way are pressed together the friction thus produced causes the revolution of the one wheel to make the other wheel revolve also. But if the wheel which is being driven in this way be stopped, or encounter some very formidable resistance, no break-down whatever occurs, as the driving wheel merely glides or slips upon the other without causing it to revolve. The friction between the two wheels must be rather greater than that required to work the cane mill under normal circumstances, and this of course entails greater motive power. Break-downs to mill machinery caused by jamming of rolls may be avoided by an arrangement depending upon quite a different principle entirely. Under ordinary conditions the distance between the rolls is adjusted by screws, but if one of the rolls could be pushed towards the other with the requisite force there would be no need for screws. This is what is done in the case of the roll B, which is pressed towards the roll C by great pressure generated by levers and weights. Any very unusual strain upon the roll is, however, sufficient to overcome this pressure which urges B towards C. When this takes place, therefore, the roll B is pushed back from C, and the obstacle causing the obstruction is thus allowed to pass through.

*Influence of speed of rolls on percentage yield of juice.*—A larger percentage yield of juice is extracted from the cane when the speed of the rolls is about two to three revolutions a minute than when driven at greater speed. Looking to the vast mechanical power brought to bear upon the cane in its passage through the sugar mill, it might be imagined that almost the whole of its juice would be expressed. Although great improvement has no doubt taken place of late years, yet not so very long ago not more than 50 per cent. of the juice was extracted on an average. The causes of such a deficiency were many and varied: some of these were due to inherent defects in the construction of the mills themselves, others to imperfect handling, whilst still others were due to sheer prejudice. The good or bad working—that is, the greater or less efficiency of a mill for crushing canes—will obviously depend, other things being equal, on the distance between the rolls. As the effect of the wear and tear of continued working, the distance between the rolls naturally tends to increase; and notwithstanding that this distance may be regulated by screws and bearings, so that the rolls may be brought as near to each other as may be necessary, yet this opera-

tion involved so much time and trouble that it was very often neglected. The great point to be attended to in maintaining the efficiency of a sugar mill is in seeing to the regularity with which it is fed. The canes should be fed into the mill in an even, uniform layer, otherwise the canes, being fed into the mill unevenly, will be unevenly and irregularly pressed. The canes in the thick portion of the layer will receive great, if not undue, pressure, whilst the thin edges, if they do not escape pressure altogether, will at least not be pressed to the extent they would be had the mill been fed in a rational way. The point now insisted upon was one of the great objections to the old-fashioned vertical rolls. These vertical cylinders had to be fed much more carefully than the horizontal rolls. But however excellent the sugar mill may be, and however perfect may be its working condition, and however carefully and punctiliously it may be fed, one great cause of imperfection still remains. This is due to inherent defects in the mechanical principles on which the sugar mill is constructed. From very obvious mathematical deductions it will be readily granted that the line of maximum pressure between the two proximate revolving rolls (cylinders) is infinitely small in width. The width of the layer of cane at one moment subjected to this pressure must, therefore, be infinitesimally small. But if the woody fibres and cellular tissue composing the sugar cane had no elasticity, there would be but little force in this line of argument. But the cane is comparatively rather elastic; it, as soon as it passes over the line of maximum pressure, expands, whilst at the same time this expanded portion acts exactly like a sponge and again sucks up a portion of the juice as soon as it has been expressed. This was a defect inherent to the principle of construction of the mill, which could not be altered so long as that principle was adhered to.

*Bessemer's machine for expressing the juice from the sugar cane.*—With the view of obviating this defect inherent to roller mills, and with the object of more completely extracting the juice from the cane than is possible in any mill built on the roller principle, Sir Henry Bessemer invented a machine for expressing juice from sugar canes by means of plane contact—that is, by the pressure of two flat surfaces like a hammer on an anvil. This machine when in action performs two separate functions: it not only cuts the cane into short lengths, but squeezes them at the same time. A diagram of the principle on which this mill works is shown in Fig. 86. P is a solid plunger which oscillates to and fro in a tube. The plunger is driven by a crank, and receives considerable power with the aid of the inertia of a fly-wheel. The canes are supplied to the mill by the vertical tubes A and B. The plunger in the position shown in the figure is about to move towards D. In its passage it cuts off the end of the cane in the tube B, and compresses the segment thus

cut off against the mass of bruised cane D. The juice which flows from the cane escapes through the holes H H in the tube. These holes are conical, with the narrow end inside to avoid clogging. On the return of the plunger to C a segment from the end of the cane in A is cut off, and has its juice expressed. A new length of cane in B descends into the tube to be ready for a fresh operation. Thus two canes are supplied to each tube at the same time, and several plungers and tubes can be worked by the same engine. It is believed that by this process the juice is more completely expressed than by rolling. Not only is the sugar saved, but the expressed canes (bagasse) are drier, and therefore more fit for fuel; and this is a very important consideration when fuel is scarce and expensive.

*Exhaustion of the bagasse.*—The bagasse from the ordinary mill still contains 25 to 30 per cent. of juice. It is drenched with water and crushed again. The bagasse conveyor therefore passes through

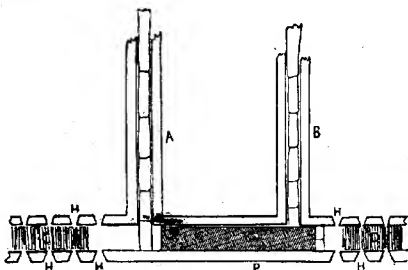


FIG. 86.—Diagrammatic representation of Bessemer's machine for forcing the juice from the sugar cane.

the bottom of a flat tank filled with water, and brings up the moistened bagasse to pass under a new mill, consisting often of only two superimposed rolls (Fig. 82), but sometimes under a similar mill to the first. The bagasse is moistened and milled twice so as to leave as little sugar as possible.

*Shredders.*—The shredder which is sometimes used, e.g., *Fauve's* shredder (Fig. 83), is a mill with grooved rolls turning at different speeds. The cane is passed up under its efforts in an inverse direction, and is more easily pressed and moistened. A shredder invented by *Baze* consists of a steel arm passing between the bars of a strong grating so that the cane falling underneath its arm is hashed up into small pieces, whilst still retaining the form of fibres long enough to yield a good pressing and capable of being easily moistened.

*Systematic mashing of the bagasse—Perichon's process.*—This' pro-

cess is adopted in the factory of Rodah, Egypt, belonging to the Daira Sanieh of the Khedive. Its object is to extract the sugar left in the bagasse produced by the passage of the cane, either through a single mill or through two consecutive mills without imbibition between the two crushings. The bagasse after one or two crushings falls on a conveyor, which carries it into the washing tanks, mounted on wheels and so arranged that the liquid can flow out as quickly as possible and the exhausted bagasse can be discharged without difficulty. Each washer has therefore a wide discharge valve in its



FIG. 87.—Systematic levigation of the bagasse. Perichon's process as used in Egypt (CAIL).

lower part under a perforated wrought-iron false bottom, and at a convenient height, a movable screen, removed when filling in or discharging the bagasse, and fixed to the sides of the tank to keep the bagasse at a uniform height during the systematic exhaustion. The wash-tanks containing the exhausted bagasse are run in succession on to a hydraulic bascule, and after removing the movable screen they are tilted (Fig. 87) on to a conveyor, which carries the bagasse to a crushing mill to remove the greater part of the water, so as to enable it to be afterwards used as fuel.

Hot water is run into the battery of wash-tank tilting waggons,

the number of which depends on the density desired in the wash-liquors, the nature and richness of the bagasse, and the exhaustion aimed at in the wash-tank whose bagasse is most exhausted. The wash-water passes in succession through the wash-tank tilting waggons, and becomes richer in sugar as it passes from the one to the other, until it reaches the last, filled with fresh bagasse as it comes from the mills, so as to form the wash-juice, which is sent to the factory to be treated either by itself or in admixture with natural cane juice. The battery being in full working order, when the time comes to remove the wash-tank tilting waggon containing the exhausted bagasse, the liquid in which the bagasse is immersed in the different tank waggons is previously and simultaneously emptied and pumped into as many calorisors on an upper floor as there are wash-tanks in the battery. The entire battery is then shifted back by the length of the suppressed wash-tank in the rear, and a new tank charged with fresh bagasse from the mills is run on in front. Then the wash-liquors previously removed and reheated in their respective calorisors, where they have been methodically treated with lime, are run back into all the wash-tanks of the battery. These dischargings, shiftings, and filling of the battery are repeated each time that a wash-tank containing exhausted bagasse is withdrawn from the front of the battery and replaced behind by another charged with fresh bagasse. Perichon's process has been used during three seasons in the Rodah factory. It consists of two batteries each of eleven wash-tank tilting waggons for treating the bagasse coming from two successive crushings without imbibition between the two pressings; and it gives every satisfaction, both by simplicity in working and by the results obtained, which are much superior to those previously obtained in the same factory by re-pressing after imbibition. In fact, in Rodah factory, which in 1900-1 treated 1,160 tons of cane per twenty-four hours for ninety-six days, there was obtained a total extraction of mixed natural juice and washed juice of 89.53 litres only per 100 kilogrammes of cane, and the average density of the wash-juice at 15° C. was 1.043 (6°·2 Baumé), whilst this average of the natural juice at 15° C. did not exceed 1.068 (9°·45 Baumé). The yield was—

	Per Cent. of Cane.
Sugar No. 1, polarising 98.50 . . . . .	10.17
Sugars Nos. 2 and 3, which were not yet turbinéd, polarising about 84 (estimation) . . . . .	0.60
Final molasses, polarising about 84 (estimation) . . . . .	2.80

The average richness of the cane was 12.2 per cent. of sucrose, and sucrose left in the exhausted bagasse was about 0.4 per cent. of the cane treated. This process may be used with fixed batteries as



well as movable batteries like those of Rodah. The Directors, satisfied with the results obtained at Rodah, decided to use the process in the season of 1901-2 in his Bibeh factory, where the bagasse from 1,800 tons cane would be treated daily. Increase in the density of the wash-juice from the different wash-tanks which compares the two batteries installed in the Rodah factory:—

Left Battery.		Right Battery.	
No. of Wash-Tank.	Density at 15° C. ° Baumé.	No. of Wash-Tank.	Density at 15° C. ° Baumé.
1 exhausted bagasse	0.4	1 exhausted bagasse	0.45
2 " "	0.8	2 " "	1.0
3 " "	1.3	3 " "	1.6
4 " "	1.9	4 " "	2.0
5 " "	2.7	5 " "	2.7
6 " "	3.3	6 " "	3.3
7 " "	4.0	7 " "	3.9
8 " "	4.6	8 " "	4.7
9 " "	5.4	9 " "	5.6
10 " "	6.2	10 " "	6.1
11 final withdrawal of juice . . .	6.8	11 final withdrawal of juice . . .	6.8

*Bagasse as fuel.*—The final bagasse is used to fire the boilers, in which it burns like straw *when it is dry*. But this drying is not always easily accomplished. The rind of the sugar cane, like most grasses, contains much silica, so that when it is burnt a glassy slag results which renders the crushed cane ill adapted to serve as fuel. Spread on a field in very hot countries with no rainfall the sun soon dries it. But, in countries like Louisiana, with a rainy climate, the sun often not to be seen, the bagasse has to be stored in sheds, a dangerous practice, as the heat generated by the fermentation is liable to cause conflagrations. Moreover, it is very costly, seeing the enormous volume occupied by the bagasse from a few days' working. Otherwise, the bagasse has to be burnt in the wet condition, after pressure in the last mill. But this wet bagasse does not burn well unless it be dried in the furnace itself, where it is by special arrangements exposed to the heat of the flame before being burnt. But such a fuel produces but little steam. Nevertheless, as it is the only way of getting quit of it, except as the so-called *molasscul*, this has to be put up with, and the small number of calories generated by it in the boilers taken as much advantage of as possible.

The furnaces for burning wet mill bagasse are numerous; but diffusion bagasse is altogether different.

*Furnace for burning green bagasse.*—The accompanying illustration (Fig. 88) shows a furnace for burning green bagasse as it comes from the mills, with an inclined grate or hearth. Sometimes

...ating grate is used, so as to let the ashes fall through as soon as formed, thus reducing hand labour. As on many extensive

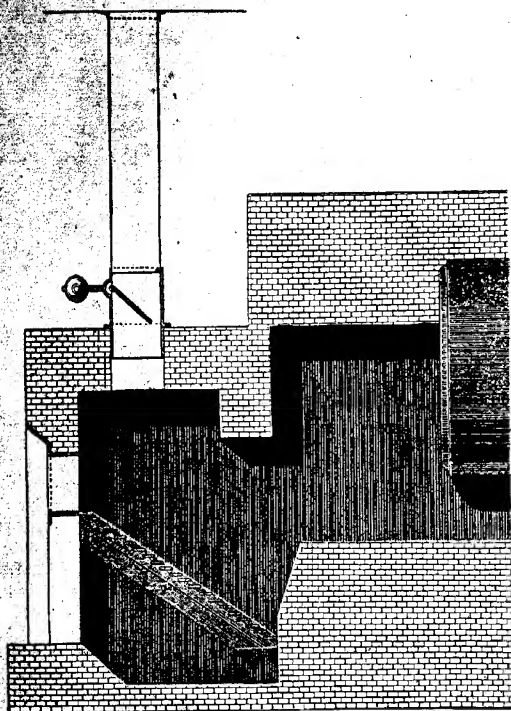


FIG. 88.—Furnace for burning green bagasse (CAMBRAY).

plantations green bagasse is unfortunately the only available fuel, the question of the best form of furnace is important.

## CHAPTER IX.

### SUGAR-CANE DIFFUSION—THE RESULTS OBTAINED ON THE LARGE SCALE BY THE UNITED STATES DEPARTMENT OF AGRICULTURE AT MAGNOLIA, LOUISIANA, U.S.A.

THE principle of diffusion is applied in *favourable localities* to the extraction of sugar from the sugar cane as a natural sequence to the success obtained with the sugar beet (Chapter IV.). A cane-sugar diffusion battery is altogether similar to a beet diffusion one. The only difference is in the shape of the diffusers (Fig. 89), which should be truncated, the largest base underneath. For evacuation purposes, the discharge door occupies the whole of the bottom. The diffusers are heated (Table XLVIII.) to a much higher temperature than the European beet diffusers, and the diffusion process is carried on under altogether favourable conditions, because it is not really a diffusion process—it is simply a washing of the slices, which immediately part with their saccharine juice.

*The Magnolia diffusion experiments.*<sup>1</sup>—The results of the diffusion work at Magnolia, Louisiana, U.S.A., though unsatisfactory in some respects, *thoroughly demonstrate the practical manufacturing value of the process as applied to sugar cane.* The cane, says Mr. Spencer, will submit to rougher treatment in the diffusion battery than the beet, and consequently the manipulations are simpler. This very property of the cane often tempts the battery-man to careless work, resulting in loss to the planter. Every possible precaution should be taken to secure regularity of work. It should be remembered that the battery-man is placed in a responsible position, and he should be remunerated accordingly. *Delays incident to the diffusion battery were of rare occurrence.* With a satisfactory cane-cutter there is little probability of delays, except from bad weather. The results of 1888 season's work indicate the possibilities of diffusion, and justify a rapid introduction of the process.

*The milling of exhausted chips for use as fuel.*—On another page attention is called to the probable effect of high temperatures on the cane, especially in regard to the subsequent milling of the

<sup>1</sup> Quoted as authentic results of *actual working only.*  
(254)

exhausted chips. In 1887 the chief milling experiment was practically a failure; whereas in 1888, on the contrary, it was successful. No special adjustment of the mill was made for the experiments in either case.

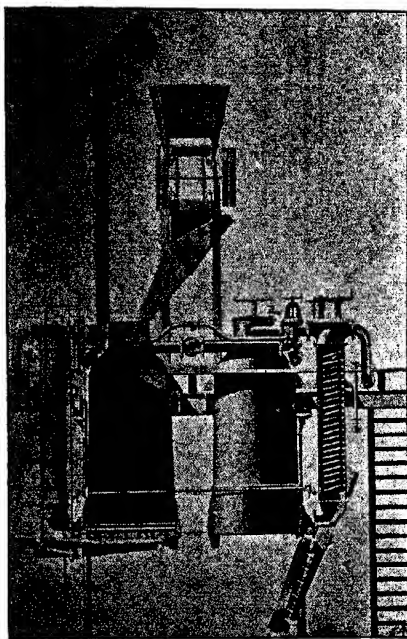


FIG. 89.—Cane-sugar diffusers, showing elevator, feed-chute, and calorimeter, etc. (C&L).

The following percentages of water were left in average samples from each mill in 1888 :—

- (1) Chips from three-roller mill retained 60·85 per cent. water.
- (2) " " five-roller " " 52·65 " "

The chips burned freely; those from the three-roller mill would probably have burned fairly well, but no test could be made.

The milling and burning of the chips is not so difficult a problem as usually considered. It would be much more economical than the present practice at Magnolia of dumping them into the Mississippi.

*Coal consumption in sugar-cane diffusion working.*—The value of the figures which Spencer obtained in his diffusion work at Magnolia was very much lessened by the adverse conditions under which steam was generated in the bagasse boilers. The bagasse chute (Fig. 88) was arched over with brick, and coal was used for firing. Economical steaming was out of the question under these conditions. The bagasse burner was built under the Fiske patents. The boilers were cylindrical. In the rest of the steam plant the boilers were of the double-flue type, for burning coal. The figures are *not given as an exhibit of economical management, but are simply a statement of actual work.* The total coal consumption for the diffusion work was 2,674,585 lb. This includes all the coal used, except in bone-black room. The fuel burned in swing-out sugars after the close of the season is divided between the mill and the diffusion work in proportion to yield of sugar. The only basis for estimating the coal consumption for each run is the relative actual dilution (see p. 269).

TABLE XLVI.—RÉSUMÉ SHOWING THE COAL CONSUMPTION FOR EACH RUN.

Period.	Coal Consumed per 1,000 lb. Sugar.	
	Lb.	Barrels of 180 lb.
Third run . . . .	1,942	10.79
Fourth run . . . .	2,029	11.27
Fifth run . . . .	1,757	9.76
Average for the season . .	1,875	10.42

In examining the coal statement, work must be taken into account. It is safe to estimate very nearly as large a fuel consumption during irregular work as when the house is working to its full average capacity. Diffusion at Magnolia more than doubled the coal bills. Taking delays into account and failure of cutter to furnish sufficiently thin chips for work at low dilution, a lower consumption of coal could hardly have been expected. Taking no account of the exhausted chips, which ought to furnish a large proportion of the fuel, it is conservative to estimate a coal consumption of from 1,200 to 1,400 lb. per 1,000 lb. of sugar as sufficient under favourable conditions.

*Difficulties encountered in sugar-cane diffusion.*—The great diffi-

culty in sugar-cane diffusion work is to find a cane-cutter. That generally used consists of a revolving plate like that used for beets, but the knives are simple plane-cutting blades. Above the revolving plate are six peculiarly shaped hoppers, fixed in such a way that the cane fed into them presents itself in front of the knife under a certain angle. The cane is cut on the slant, and not perpendicular to the axis. Rounded, elongated slices are thus obtained, which fall into the diffusers from a chute in the same way as in the case of beets.

*Sangerhausen cane cutter.*—This cutter consists essentially of a horizontal disc carrying twelve knives set parallel to the radii of the disc, and revolving in a cast-iron shell. There are six cane chutes or hoppers placed at an angle of 45 degrees to the surface of the disc. The cane, thrown lengthwise into the chute, descends by gravity to the knives, where it is sliced diagonally. A suitable arrangement for throwing the chips into the elevator boot is provided. This cutter was designed by the constructors to revolve 110 revolutions per minute, and its capacity was guaranteed to be from 200 to 250 tons of chips per twenty-four hours. After overcoming numerous and serious faults in the construction of the cutter and increasing its speed to 180 revolutions (an increase of 63 per cent.), 195 tons of chips were cut from straight cane in twenty-four hours, the largest day's work accomplished at Magnolia. An able and experienced sugar manufacturer had charge of this work, and it was possible by his skill to overcome the difficulties in preparing the cane for the battery. After the first failure of the cutter, Dr. Wiley ordered small steel scrapers to be attached to the upper surface and side edge of the cutting disc. The cane was very juicy in 1887, hence he was enabled to cut nearly 1,000 tons of cane without appreciable wear of these scrapers. In 1888, on the contrary, the cane was exceptionally woody, and *scrapers of the best file-steel were worn out in cutting less than 400 tons of cane.* In addition to the trouble with these scrapers, it was found that the fibres from the cane collected between the disc and outer shell and soon completely blockaded the cutter. Openings were cut both in the cover and upper part of the shell to relieve the disc of the accumulations of fibre. It was only then that it was possible to use the cutter at all. Economical cane diffusion demands an exceedingly thin slice or chip. At Magnolia it was impracticable to obtain a chip less than an eighth of an inch. This is double the thickness required by good work. Owing to the large labour bills, the difficulty in regular adjustment of the knives, and impossibility of obtaining a sufficiently thin chip, the cutter, says Spencer, is not suitable for diffusion work in Louisiana.

*The Hughes' cane cutter.*—The system of cutting cane used at Colonel Cunningham's estate in Texas, and in the sorghum houses

in Kansas, is the invention of H. A. Hughes, of Cape May City, N.J. This cutter consists of a metallic cylinder, carrying a number of knives whose blades project from the circumference of the

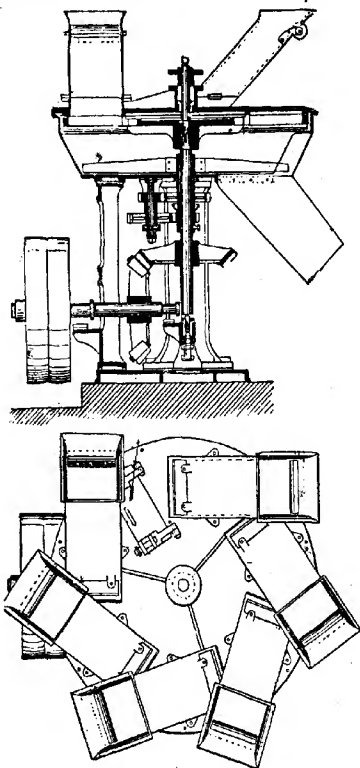


FIG. 90.—Cane cutter (CALL).

cylinder in the direction of rotation. The cylinder is rapidly revolved in front of a dead knife set parallel to its face. It is always used in connection with an ensilage cutter, which latter furnishes the short pieces of cane which are thrown into a hopper, where they

are caught by the knives and carried against the dead knife. A small piece of cane is cut off and carried between the knife and the dead knife, and by the centrifugal force is thrown into a receiver below.

The rapid advancement of cane diffusion is largely due to Mr. Hughes' successful cutting apparatus.

*The U.S.A. national cane shredder.*—The adaptation of the cane shredder to the preparation of cane for the battery was suggested. This machine was used several seasons by Governor Warmoth in the shredding of whole cane for the mill at Magnolia. If its work during this time can be taken as a criterion the shredder could, in Spencer's opinion, be readily adapted to the requirements of diffusion.

*Details of sugar-cane diffusion work. The diffusion battery.*—Spencer gives the following details of the diffusion battery used in

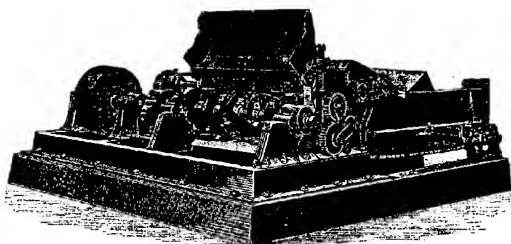


FIG. 91.—Cane cutter.

his experiments on the large scale at Magnolia. It was built in 1887 by the Colwell Iron Works of New York. It was enlarged in 1888 by Edwards & Hauptmann of New Orleans, according to the directions of Governor H. C. Warmoth.

*The number of cells in the battery.*—In the enlargement of the battery the only changes made were in the length of the cells and calorimeters or heaters, and the addition of two new cells. The battery as used consisted of 14 cells, arranged in a circle, and charged from a central reservoir by means of a revolving chute (see Fig. 89, p. 255). Governor Warmoth has since substituted a double-line battery for the circular battery, and a Rillieux evaporator for the Yaryan which it is claimed works better.

*Dimensions of the cells.*—The cells were 11 feet long by 44 inches in diameter; the nett cane space 107 cubic feet; the upper doors 30 inches in diameter, and the nett opening at the discharge door 44 inches, the full diameter of a cross-section of the cell. The



joint between the discharge door and the bottom of the cell was the ordinary hydraulic closure (p. 52).

*Calorisators.*—The calorisors or heaters (Fig. 89), as originally constructed, were 49 inches long by 2 inches in diameter in each, giving a heating surface of 17.1 square feet. In enlarging the battery, seven tubes,  $1\frac{1}{2}$  inches by 41 inches, were added, giving an additional heating surface of 9.2 square feet per calorisor. The heating surface was sufficient for the work, but it would have been a wise precaution to have increased it considerably more. The heating surface per cubic foot of cell space was  $0.246$  square feet, or nearly 4 cubic feet per square foot of heating surface.

*Juice and water mains, etc.*—The juice and water pipes were of cast iron, and have a nett diameter of 4 inches. The compressed air-pipes were  $1\frac{1}{2}$  inches in diameter. The accumulator for compressed air had 75 cubic feet capacity. A 2-inch main furnished ample steam for the battery.

*Method of removing exhausted chips.*—A circular track under the cells, provided with a flat car having its axles fixed in the direction of the radii of the circle, served to carry the chip car from cell to cell. The flat car was fitted with a piece of track of the same gauge as that of the permanent railroad leading to the river. When a cell of exhausted chips was discharged into the car, the flat car was drawn by a mule to a point opposite the main line and the chip car run off and taken to the river to be emptied. The round trip required less than seven minutes. A large flat-boat projecting into the river served to carry the track far enough out for the current to wash the chips away.

*Circular batteries versus line batteries.*—The question of arrangement of a diffusion battery will generally depend upon local conditions. The batteries built for the U.S.A. Agricultural Department previous to that at Magnolia were of the type known as line batteries. The circular arrangement was selected for Magnolia in order to give the planters an additional example of the different types of diffusion batteries. The circular arrangement has many advantages. It also has disadvantages with which the line battery is not compelled to contend. The principal of these latter is the difficulty attendant upon the removal of the exhausted chips. A builder of this class of machinery informed Spencer that there is no difficulty in arranging to move the chip car from cell to cell by power and finally run it outside the building for dumping. A circular battery, says Spencer, possesses decided advantages over all other forms in ease and regularity of charging the cells with cane chips, neatness of arrangement, and facility of controlling the work. The valves should be so arranged that they can be manipulated from inside the circle. The measuring tank should also be placed inside the circle, preferably at the centre. *The chip*

*chute*.—The chute (Fig. 89) should be entirely supported from above, a counterpoise relieving the strain caused by the weight of the chute coming entirely on one side. Instead of a sliding door, to block the flow of chips when moving from one cell to another, the end of the chute should be provided with a hinged spout, balanced in such a manner that it can be thrown back and stop the flow of chips, the bottom of the spout becoming a gate. When this arrangement is adopted, there is then ample room to place the measuring tank in the centre of the upper platform. The valves and pipe-lines being on the inside of the circle, the battery-man has easy control of the work, and cannot be pardoned for overheating the cells or making other errors. In the enlargement of the battery the size of the pipe-lines was not proportionately increased. It was found for rapid work—i.e., a cell every seven and one-half minutes—that a pipe area of 12½ inches is not quite sufficient, but 20 inches would be ample for a cell of the dimensions of those at Magnolia. The calorimeters or heaters were of sufficient capacity. In enlarging the calorimeters the original outlets for water of condensation were retained. *Control of diffusion work*.—In order to arrive at comparable results, and place the records beyond the possibility of error through neglect or forgetfulness of the workmen, some automatic device for registration is essential. These records, the work of an instrument, made entirely without prejudice or fears of punishment for negligence, become valuable data for locating and correcting errors. The first cost will be many times repaid. A battery-man, no matter how faithful and capable he may be, is liable to make errors that may prove very expensive, and render studies to improve the work valueless. *The diffusion of sugar cane presents many conditions quite different from those which exist in the beet*. The comparatively small amount of work done in the diffusion of cane in the U.S.A., and many of the conditions under which it has been done, have prevented a careful study to determine the most favourable conditions for such work. Now that the success of the process is fully demonstrated, attention should be given to improving the work of the batteries. In addition to the automatic records of Horsin-Deon's and Eugene Langen's apparatus (see Fig. 31, p. 84, and Fig. 33, p. 87), the following further checks on the battery work were adopted. All the automatic registers were kept under lock and key, and out of sight of the battery-man. This workman was provided with a checking system that promptly notified him of errors.

For this purpose, blanks, ruled as in Table XLVII., were furnished the men at Magnolia.

The men were required to fill in the blanks as in Table XLVII. and enter on the back of each sheet the cause of delays. The most frequent error is drawing two or three times from one cell. An

immediate fall in the density of the juice notifies the workman of his error. The failure of the cell number to correspond with the number automatically registered notifies the chemist or superintendent of the error. To illustrate the above-mentioned error is appended a transcript of the battery report for two watches, December 9. The numbers in the column headed "Temperature" indicate the temperature of the juice at the time of determining its density, and not necessarily at the time of drawing the charge. It will be noticed in this report that the density of the juice began to fall rapidly at 5.48 P.M.

TABLE XLVII.

MAGNOLIA PLANTATION.									
Date.—December 9.					Watch.—Second day and first night.				
Battery-man.....									
Cell No.	Time when Drawn.	Density.	Temperature.	Litres Drawn.	Cell No.	Time when Drawn.	Density.	Temperature.	Litres Drawn.
		°Baume.	°C.				°Baume.	°C.	
4 <sup>1</sup>	1.21	6.0	38	1360	13	7.45	5.8	37	1360
5	2.06	6.1	37	1360	14	7.54	5.8	38	1360
6	2.15	4.6	50	1360	1	8.04	5.7	43	1360
7	2.25	5.3	38	1360	2	8.15	5.4	48	1360
8	3.34	5.2	37	1360	3	8.24	5.5	45	1360
9	3.49	4.2	50	1360	4	8.33	5.4	47	1360
10	4.12	5.0	40	1360	5	8.42	5.5	47	1360
11	4.22	5.0	39	1360	6	8.53	5.5	48	1360
12	5.21	4.3	50	1360	7	9.02	5.6	46	1360
13	5.30	4.9	55	1360	8	9.11	5.7	46	1360
14	5.39	3.4	57	1360	9	9.20	5.7	46	1360
1	5.48	3.3	50	1360	10	9.29	5.8	46	1360
2	5.57	2.0	58	1360	11	9.38	6.0	43	1360
3	6.06	1.8	57	1360	12	9.59	6.1	43	1360
4		Not drawn.			13	10.08	5.6	50	1360
5	6.25	3.9	44	1360	14	10.17	5.6	49	1360
6	6.35	4.2	40	1360	1	10.34	5.6	49	1360
7	6.45	4.5	41	1360	2	10.44	5.2	52	1360
8	6.58	5.0	37	1360	3	10.55	5.1	52	1360
9	7.07	5.2	36	1360	4	11.14	5.4	46	1360
10	7.16	5.5	36	1360	5	11.23	5.6	46	1360
11	7.25	5.6	38	1360	6	11.43	6.0	40	1360
12	7.35	5.8	37	1360	7	11.53	5.4	46	1360

<sup>1</sup> Work very irregular during second watch, 1.21 to 6 P.M., on account of trouble with the cane cutter.

This sudden fall in density is due to more than one draw being made from one cell, *i.e.*, the workman neglected to close a certain valve connecting with the juice main, and hence, instead of drawing from the cell last filled with fresh chips, drew repeatedly from a preceding cell through this neglected valve. The battery-man coming on duty the first night watch detected the errors from his predecessor's report, and corrected it. Considerable irregularity in the recorded density of the juice is due to great variations in the temperature at which the reading was made. It is perhaps needless to add that the battery-man who made these errors was relieved from duty the following day when he carelessly repeated the above mistakes. It would be easy for a workman to conceal his error by making a false entry in his report. The use of the automatic registering apparatus would effectually prevent or detect such false entries.

*Delays due to encrustation on Yaryan.*—The diffusion battery having been used three days continuously, it was decided to clear the yard and sugar house and begin test runs. These runs began December 1, and were continued until the end of the season. In this time there were few delays chargeable to the battery. There were numerous delays caused by the inefficiency of the cutters and the extremely foul condition of the Yaryan quadruple-effect (p. 175). This latter failed to work up to its guaranteed capacity on account of a thick deposit of scale on the tubes. Late in the season Mr. Yaryan visited the plantation, and recommended boiling out the pans with caustic soda. This treatment was very effective, and the capacity was soon amply sufficient for the work required. In preparation for further enlarging his sugar house, Governor Warmoth contracted for an 18-coil quadruple-effect of the Yaryan system. But a Rillieux evaporator has, it would appear, been substituted for the Yaryan since 1901. Some considerable delay was caused at the beginning of the season on account of the clarifiers not being in readiness. It was Governor Warmoth's intention to depend entirely on the clarification of the juice in the diffusers. This work was unsatisfactory, so he returned to the ordinary method.

*Manipulation of the diffusion battery.*—Fill two or three cells with water heated to near its boiling-point. Let these cells precede cell No. 1, *i.e.*, the first cell filled with fresh chips. For convenience of reference the cells are referred to in numerical order, invariably calling the one in immediate connection with the water supply No. 1, and that containing fresh chips (after the first round of the battery) No. 12. No. 13 is open for the discharge of exhausted chips, and No. 14 is filling with fresh chips. By a proper manipulation of the valves force water into the first of the cells containing hot water, driving the water forward and into No. 1 at the bottom. By admitting the water at the bottom of the cell, the air is driven

out at the vent in the cover. In the meantime No. 2 is filled with fresh chips. When No. 1 is full of juice the valves are changed, and the circuit established through the valve connecting with the upper part of the diffuser. The valve connecting with the bottom of No. 2 is then opened, and the juice from No. 1 passes in at the bottom of this cell, water taking the place of this juice. No. 3 is filled with chips, and the same operations are repeated, and so on, until six or seven cells are filled. The number of cells so filled is dependent largely on the temperature of the water entering No. 1 and the probable extraction. Let us assume that seven cells have been filled. A charge of juice must now be drawn. The juice having passed through seven cells of chips, no draw having been made, has about reached its maximum density. The work is now continued, a charge of juice being drawn from each cell filled. When No. 12 is reached, the hot water in No. 13 is discharged into the ditch; while No. 13 is filling, the water in No. 14 is discharged. The first round of the battery is now completed. The chips in No. 1 have been treated twelve times with fresh water and are now ready to be rejected. While No. 14 is filling with fresh chips the exhausted chips in No. 1 are being removed. This routine continues without variation. A few hours' practice at a battery is sufficient to train an intelligent labourer to do this work.

*Influence of the dimensions and form of the cell.*—If we place cuttings of cane in a vessel and surround them with water, no matter what may be the size or shape of the vessel, an equilibrium will soon be established, and the diluted juice bathing the chips will be sensibly of the same density as that contained in the cuttings themselves.

If, in the construction of a diffusion cell, we give it a diameter of 4 feet and a depth of but a few inches, there is no reason why the extraction should be either better or poorer than in a cell a few inches in diameter and several feet long, provided the circulation is equally good in each case. It is this proviso which should control the dimensions and form of a diffusion cell, and not the possibility of an increased or diminished extraction through variations in length of the column of chips which the water must traverse. The length of the column of chips has no influence whatever upon the extraction, but should not be sufficient to impede the circulation. In the manufacture of sugar from beets there is a serious objection to a large cell, hence the tendency to make a capacity of 300 tons per day per battery a limit. This objection is the liability of the beet cuttings packing or matting, and thus interfering with the circulation. In the diffusion of cane even at high temperatures we find no such tendency to matting. The extraction in the Magnolia battery this season was very uniform, notwithstanding the increased length of the cells. In cells of large diameter there is a possibility of diffi-

culty in uniformly distributing the juice. The experience in cane work has been so limited, and so few batteries have been erected where a careful study of the work has been made, that we have little data on this point. A cell of small diameter can be built for less money than one of the same cubical contents but greater diameter. This refers especially to large batteries. The lower doors of a cell of large diameter should have more than two supports, namely the hinge and latch, in order to prevent springing. If the length of the cell is excessive the great length of the column of cane chips will retard the current of juice, and it will be necessary to increase the water pressure. The essential conditions in the construction of a diffuser are that the form and dimensions must be such as to secure the best circulation of the juice through the chips.

*Clarification by liming in the diffusion battery.*—The first few days of the season 1888-89 at Magnolia, an attempt was made to use lime in the cells to neutralise the acids of the juice. The temperature of the three cells preceding that containing the fresh chips was maintained at as nearly  $95^{\circ}$  C. ( $203^{\circ}$  F.) as possible. The results may be summed up briefly as follows. The diffusion juice was bright and perfectly clear. To verify that the clarification was complete, the juice was run into the clarifiers and heated to the boiling-point. Quite a "blanket" formed, considering the previous preliminary clarification. These impurities evidently resulted from the partial clarification that had taken place in No. 12. The fresh chips, being very much colder than the juice coming from the preceding cell, lower the temperature below the point to a good clarification. At the beginning of this work it was suggested to draw from the second cell from the last, i.e., No. 10. The juice from this cell, having been heated to the highest temperature practicable in the battery, is thoroughly clarified. This plan was not adopted, since it reduces the number of cells under pressure to 10, and necessitates driving two cells of juice ahead. The juice in these cells soon reaches its maximum density, and serves to heat the chips to such a temperature that a good clarification can be obtained. An attempt was made to heat the chips in No. 12 in the following manner. It was filled with chips and juice in the usual way, except that compressed air was used to force the juice through the cells without changing the main battery valves, the air vent on No. 1 was opened, that on No. 12 closed, and the current reversed, forcing air into No. 12. The air vent on No. 12 was again opened, and the cell filled with juice, as usual in regular work. It may be seen that the chips in No. 12 were twice bathed in hot juice, the first time raising their temperature considerably, and the second sufficiently high for clarification. The draw was made from No. 12 as usual. This method of working, although it accomplishes the desired result, was too complicated, hence was not adopted. Fromentin advises

*"the use of a small quantity of lime in the diffusion of beets, two to three litres of milk of lime at 25° Baumé being added per diffuser. An increase in the purity of the juice and a better extraction are obtained."* Fromentin cites an experiment in which the purity of the juice was increased 2 degrees. A complete clarification of the juice was not claimed. Subsequent treatment by the carbonation process was necessary. In 1883 O. B. Jennings, of Honey Creek, Wis., was granted a patent for certain processes in sugar manufacture, in which he uses either dry lime or lime whitewash mixed with the cane cuttings for the purpose of obtaining a thorough defecation in the diffusion apparatus. Mr. Jennings also claims that this process is applicable in the diffusion of sugar cane. Lime was used in the diffusion cells at Wonopringo, Java, in 1887, for the purpose of clarification. Lime was also used in the diffusers at the Planters' Experiment Station, Kenner, La. Dr. W. C. Stubb's experiments with this process, which were made entirely independent of the work of other experiments, were very successful. This method of clarification is discussed in U.S.A. Bulletin No. 20, Division of Chemistry. A few experiments were made at Magnolia, on a small scale, to determine how perfect a clarification can be obtained by this process. A pressure flask, such as is used in analytical work, was nearly filled with cane chips; sufficient lime was added to neutralise the acids in the juice, and the flask was finally filled with clarified diffusion juice, closed, and heated ten minutes to a temperature of 95° C. (203° F.). The flask was cooled, opened, and the juice was filtered through linen cloth. The filtered juice was then heated to its boiling-point in the open air. It remained perfectly clear, and even on boiling did not show signs of turbidity. The conditions of this experiment were the same as those existing in regular diffusion work, except that in the latter case it is impossible to heat the last cell to as high a temperature as that obtained in the experiment. This experiment shows that, as soon as we obtain some simple method of carrying the juice in the last cells of the battery to a sufficiently high temperature, we can obtain a clarification superior to that obtained in the ordinary manner. The use of lime is especially to be recommended when the work is irregular or the cane damaged by frost and subsequent fermentation.

*Working temperature of the battery.*—Owing to the thick chips it was necessary to work the battery at a high temperature to obtain as good an extraction as possible with low dilution. There is decided inconvenience in working at high temperatures, due to the liability of the juice boiling in the heaters and steam collecting in the cells. It is possible for steam and air to collect to a sufficient extent to interfere with the circulation of the juice near the top of the cell. In the preliminary work with the diffusion battery the temperature of Nos. 8, 9 and 10 was maintained as nearly as possible at 85° C.

(185° F.). The juice from No. 11 was kept as hot as consistent with rapid work. The temperature of Nos. 2 to 7 inclusive ranged from about 65° to 70° C. (149° to 158° F.). With thin chips and a moderate dilution the extraction was very good. Owing to a lack of cutting capacity it was necessary to increase the thickness of the chips and work the battery at a very much higher temperature. In the early part of the work the water for the battery was passed through a large heater; later on, it was found that as good results could be obtained without the heater as when using it. The range of temperature during the greater portion of the season is shown in the following table. The battery had fourteen cells, twelve at work, one filling and one emptying. The small amount of heating surface in the caloriser should also be taken into account.

TABLE XLVIII.—TABLE SHOWING COMPARATIVE TEMPERATURE IN EACH CELL IN SUGAR-CANE AND SUGAR-BEET DIFFUSION BATTERIES.

Cell No.	Sugar Cane.		Sugar Beet.	
	Temperature.		Temperature.	
	° Cent.	° Fahr.	° Cent.	° Fahr.
1	60	140	40	104
2			60	140
3	70-80	158-176	80-85	176-185
4				
5				
6				
7				
8	95	203	65-70	149-158
9	95	203		
10	95	203		
11	85	185		
12	70	158	40-50	104-122

The low temperature of Nos. 1 and 2, 11 and 12, in the second column is noticeable. In Nos. 11 and 12 this low temperature is due to the moderately heated juice in Nos. 3 to 10 coming in contact with the cold beet cuttings. The temperature of Nos. 3 to 10 is purposely maintained at a moderate degree, to prevent the cuttings from swelling and impeding the circulation. Even if it were practicable, so far as the capacity of the calorisers is concerned, to raise the temperature of No. 1 above 40° C. (104° F.), it would be decidedly objectionable on account of the effect of high temperatures on the beet cuttings. Practice has demonstrated that a



temperature above 40° C. in this cell is liable to seriously interfere with the pressing of the exhausted cuttings. Possibly the high temperature at which we usually conduct cane diffusion is largely the cause of the difficulty that has been experienced in milling the exhausted chips (see pp. 254-6 and 267). In the experiments made in 1887 at Magnolia considerable difficulty was experienced; so much, in fact, that the experiment was practically a failure. In the battery work the water entering No. 1 was heated to about 71° C. (160° F.), and by the time it reached the second cell its temperature was little below the boiling-point. But in 1889 when the milling experiment was very successful, the temperature of the first cell did not exceed 60° C. (140° F.). Owing to the trouble with the cutter, there was no further opportunity to make experiments in conducting the battery work at different temperatures. With thin chips, however, the maximum temperature need not exceed 85° C. (185° F.). The relatively lower purity of the diffusion juice as compared with the normal juice was due to the high temperature at which it was necessary to work. Juice extracted at a temperature of 85° C. (185° F.) admits of easier and more thorough treatment in the sugar house.

*Dilution.*—Two methods of stating the dilution of the normal juice are employed in this report, namely *apparent dilution* and the *actual dilution*. In addition, the extra evaporation in terms of the diffusion juice is also given. Owing to the frequent variations in the juice content of the cane we have a variable dilution even with a constant draw. For the same reason we have a variable reduction in the percentage of sucrose in the juice, aside from irregularities of extraction. The relation of the diffusion juice drawn to the actual amount contained in the cane is the *apparent dilution*. It has been customary in diffusion work in U.S.A. to arbitrarily assume a juice content of 90 per cent. of the cane, and reduce this weight to volumetric terms based upon the density of the normal juice; a comparison of this volume of juice is then made with the volume of diffusion juice drawn. The actual volume of juice in the cane is compared with the volume of diffusion juice drawn, and the result is termed the *apparent dilution*. The nearer we approach a perfect extraction, the nearer the apparent dilution approaches the actual. The *actual dilution* is the proportion of water added to the normal juice to reduce its percentage of sugar to that of the diffusion juice, hence the actual dilution represents the evaporation necessary per cent. of normal juice to remove the added water. In calculating the dilution the sum of the percentages of sucrose and glucose (Table XLIX.) are taken, in order to diminish the errors resulting from inversion.

# SUGAR-CANE DIFFUSION

269

## RÉSUMÉ SHOWING THE AVERAGE OF MANUFACTURING RESULTS AT MAGNOLIA, 1888-89.

Cane, per cell (diffuser)	pounds	2760
" per cubic foot of cell capacity	"	25.8
Diffusion juice drawn, per cell	gallons <sup>1</sup>	339.2
" " per 100 pounds cane	pounds	107.8
" " " "	gallons <sup>1</sup>	12.3
Sucrose left in exhausted chips	pounds per ton of cane	11.28
Actual dilution	per cent. normal juice	23.4
Apparent dilution	" "	23.3
Additional evaporation	" diffusion juice	22.1
" " " "	pounds water per ton of cane	476.4
Extraction	per cent. sucrose in the cane	94.72

TABLE XLIX.—RÉSUMÉ SHOWING THE MEAN COMPOSITION OF THE NORMAL AND DIFFUSION JUICES, THE MAXIMUM AND MINIMUM DENSITY, PERCENTAGES OF SUCROSE, REDUCING SUGARS (GLUCOSE, ETC.), COEFFICIENT OF PURITY, ETC., 75 ANALYSES (MAGNOLIA, 1888-89).

	Normal Juice.			Diffusion Juice.		
	Means.	Maxima.	Minima.	Means.	Maxima.	Minima.
Degrees Brix	16.4	17.8	12.9	12.9	14.6	11.5
" Baumé	9.1	9.9	7.2	7.2	8.1	6.4
Specific gravity	1.0672	1.0735	1.0523	1.0522	1.0596	1.0464
Sucrose per cent.	14.1	15.3	10.1	11.0	12.5	8.8
Reducing sugars (glucose, etc.)	0.56	1.0	0.33	0.48	0.72	0.26
Coefficient of purity	85.97	89.50	78.27	85.27	91.88	76.47
Glucose per 100 sucrose	3.97	7.75	2.29	4.36	7.35	2.5
Inversion per cent. diffusion juice	...	...	...	0.055	2.96	...

*Increase in the juice evaporation in the sugar-cane diffusion process compared with milling.*—In figuring coal consumption in the comparison of mill and diffusion work, all statements should be based on actual dilution. In comparing figures on coal consumption of the Magnolia Diffusion Work (p. 256), Spencer remarks that manufacturers should not neglect to note the exceptional richness of the juice, and further, that in the Magnolia plantation a quadruple-effect Yaryan system was employed. The following table shows the relative quantity of water to be evaporated in mill and diffusion work. The data show an approximate average of the season's work. The estimation of the mill year is arbitrary, and is based on the previous work and the total juice in the cane.

<sup>1</sup>An American gallon of water only weighs 8 lb., but a British gallon 10 lb.

TABLE L.—EVAPORATION IN DIFFUSION WORK AS COMPARED WITH THAT OF 72 PER CENT. MILLING.

Mill Work Total Evaporation.	Diffusion Work.			
	Evaporation due to the Dilution.	Evaporation due to Increased Extraction.	Total Evaporation.	Increase over 72 Per Cent. Milling.
Lb. Water Per Ton. Cane.	Lb. Water Per Ton. Cane.	Lb. Water Per Ton. Cane.	Lb. Water Per Ton. Cane.	Per Cent.
1195.7	481.7	193.1	1870.5	56.4

The above shows an actual increase in the coal consumption for evaporation of over 56 per cent., assuming that the entire fuel supply is obtained from coal. In addition to this increase there are still others, due to a larger product, to centrifugal, and to the greater surface exposed for radiation. The increased evaporation in quadruple should not require with good boilers well fired more than 175 lb. additional coal per 1,000 lb. sugar (p. 256).

*Fuel consumption in cane and beet diffusion.*—Comparing the results with that of the best equipped beet-sugar houses employing quadruple-effects, Mr. Spencer says the latter, according to Horsin-Deon, burn 4.23 kilogrammes of coal per hectolitre of juice; comparing this with the Magnolia work, basing the figures on the same dilution, we have 100 lb. of coal consumed per average of 417 lb. in the cane-sugar house. Even the Austrian houses in their best work, where nearly twice as much diffusion juice is drawn per 100 lb. of beets as was drawn from the cane at Magnolia, burn only 180 lb. per 2,000 lb. of beets. These houses employ quadruple-effect evaporation with all Rillieux's improvements (pp. 166-172). The large fuel consumption at Magnolia cannot be charged to the Yaryan apparatus or to the vacuum pan. Repeated tests have demonstrated the high efficiency of the Yaryan. The vacuum strike pan is of the low-pressure type, and of the most modern construction. One cause of the excessive fuel consumption was, no doubt, *the use of coal under boilers designed for an entirely different class of fuel.* The fuel burned under the coal boilers as estimated by several experts was approximately 1 lb. per 6 lb. of water evaporated. There was probably a large increase due to the wastage of the waters of condensation from the battery heaters. The beet houses cited do not employ bone black, hence the fuel consumed in preparing the liquors for filtration, etc., should be deducted in this comparison. In the beet-sugar houses all the evaporation and heating of juices and syrups, etc., is

in multiple-effect. This is accomplished by the improved methods of Rillieux (pp. 166 *et seq.*). All such work at Magnolia was in single-effect except the evaporation. All the available data in diffusion work indicate that with the very best modern appliances the fuel consumption need not exceed 100 lb. of coal per ton of cane, or 500 lb. per 1,000 lb. of sugar. This is as low a coal consumption as is obtained in the best American cane houses economising wet bagasse directly in their furnaces; but if Germans and Austrians can work with this high degree of economy, cannot Americans do so as well? The day is not distant when coal will be only required as auxiliary in firing after stoppages, the exhausted chips furnishing the fuel required. Planters estimating on diffusion and intending to use evaporating appliances already in place for milling must not neglect to note that they will be compelled to work less cane per day, to compensate for the increased evaporation and extraction, *i.e.*, they must enlarge the capacity of their houses in proportion to the increased yield and dilution. In case the chips are not burned, at least two and one-half times as much coal must be provided for diffusion as would be for milling where in the latter case the bagasse is employed as fuel.

TABLE LI.—MAGNOLIA PLANTATION CROP REPORT, 1888, DIFFUSION RUNS.

	A.		B.		C.	
	Lb. <sup>1</sup>	Lb. <sup>2</sup>	Lb. <sup>1</sup>	Lb. <sup>2</sup>	Lb. <sup>1</sup>	Lb. <sup>2</sup>
First sugar (white) . . .	120,085	111.24	152,881	84.98	234,135	76.46
Second sugar (yellow C.) .	56,805	52.62	160,002	88.94	246,464	80.49
Second sugar (waggon) . .	...	...	12,037	6.69	...	...
Third sugar (waggon) . . .	31,705	29.37	71,059	39.50	114,325	37.50
Fourth sugar <sup>3</sup> (waggon) . .	21,590	20.00	35,980	20.00	61,240	20.00
Total . . .	230,185	213.23	431,959	240.11	656,664	214.45

A, third run, December 1 to 8 inclusive: cane worked, 1,079.5 tons.  
 B, fourth run, December 9 to 22 inclusive: cane worked, 1,799 tons. C, fifth run, December 23 to January 14, 1889: cane worked, 3,062 tons.

<sup>1</sup> Yield.

<sup>2</sup> Sugar per ton of cane.

<sup>3</sup> One-half of this sugar was estimated from the half swung out. Fourths are divided between milling and diffusion in proportion to the yield of the other sugars.

Crop, total . . . . .	pounds	1,855,280
Crop, average . . . . .	pounds per ton of cane	203.1
Diffusion average . . . . .	"	222.0
Mill, average . . . . .	"	167.9
Diffusion, increase . . . . .	"	54.1

The method of sampling pursued in former seasons was the only one available. 100 c.c. of juice were taken from each charge and stored in a large bottle until a sufficient quantity for a fair sample had been taken. A handful of fresh and one of exhausted chips were also taken from each cell. The former was passed through a small hand mill, and the juice so obtained was analysed, and the analysis taken to represent the normal juice of the cane. The run numbers were continued from those of the mill work which preceded diffusion. During the third run (that is, the first diffusion run) numerous experiments were made to determine the best method of conducting the battery work. The thickness of the chips soon necessitated work at high temperatures, and compelled the abandonment of further experiments. The yield of sugar in proportion to the sucrose present was larger in subsequent runs. This is partly due, in addition to richer cane, and in the fourth run a better extraction, to losses resulting from the fermentation of the syrups. This fermentation was due to two causes. The failure of the vacuum pan traps to operate satisfactorily prolonged the boiling from ten to twelve hours, five hours usually being sufficient time in which to boil a strike. This caused an accumulation of syrup in the storage tanks. At this time a supply of new bone black was substituted for the chars, which had been in use for many years. The sugar-maker never having had experience in the use of new bone black was unprepared for the difficulties in its use. (See criticism of this method of working.) The facilities for washing bone black at Magnolia were very crude, hence the char was sent to the filters badly washed. On the admission of the hot syrups to the filters dense volumes of ammonia filled the room. The syrups so filtered fermented with extreme rapidity, entire tanks of syrup showing a viscous fermentation in two or three hours. It was often impossible to centrifugal syrups grained in the pan from these syrups, hence several strikes were boiled to string-proof and sent to the hot room. This fermentation and the consequent difficulties in manufacture undoubtedly caused a large loss of sugar. The following comparison of the yield for the three diffusion runs indicates the extent of this loss:—

TABLE LII.—AVAILABLE SUCROSE (SUCROSE MINUS  $1\frac{1}{2}$  TIMES GLUCOSE).

Run Numbers.	Available Sucrose. Lb. per Ton of Juice.	Yield of Sucrose. Lb. per Ton of Juice	Available Sucrose obtained in Sugars per Cent.
Third . . .	222.7	203.63	85.66
Fourth . . .	243.3	228.73	94.01
Fifth . . .	230.8	204.39	80.56

The degree of extraction was practically the same in the third and fifth runs, the cane was considerably better in the third, the glucose per cent. being respectively 3.40 and 4.17. The necessity of apportioning the fourth sugars equally among the diffusion runs probably gives the third run a better record than it should have. The difficulty in estimating this loss emphasises the necessity of facilities for keeping the products from each run separate, if one desires a thorough chemical control. Owing to the molasses from the entire season's work being mixed in the cisterns, and several losses known to have taken place, but which could not be determined, the inversion could not be calculated for this or the other runs. The lower proportionate yield in the fifth run was probably due to methods of manufacture.

# TECHNOLOGY OF SUGAR

TABLE LIII.—RELATION BETWEEN THE DENSITY OF THE JUICE OF SUGAR CAKE AND ITS RICHNESS IN SUGAR.

(a) Degrees Baumé at 16° C.	(b) Weight of 1 Litre at 16° C. in Grammes, Density Water as 1000.	(c) Sugar per Decilitre of Juice, Lb. per 10 Imp. Gals.	(d) Sugar per Decilitre of pure Sugar Solution, Lb. per 10 Imp. Gals.	(e) Purity of the Juice.	(f) Coefficient to obtain the Sugar per Decilitre of Juice. <sup>1</sup>	(g) Sugar per Cent. of Juice.	(h) Sugar per Cent. of Cake.
7.25	1053	10.3	14.02	73	1.94	9.8	8.4
7.85	1054	10.6	14.28	74	1.96	10.0	8.6
7.50	1055	10.9	14.54	75	1.98	10.3	8.9
7.65	1056	11.2	14.80	76	2.00	10.6	9.1
7.80	1057	11.55	15.06	76	2.06	10.9	9.4
7.90	1058	11.9	15.32	78	2.06	11.2	9.7
8.00	1059	12.2	15.58	78	2.07	11.5	9.9
8.15	1060	12.5	15.84	79	2.08	11.8	10.1
8.30	1061	12.8	16.10	79	2.10	12.1	10.4
8.40	1062	13.1	16.36	80	2.12	12.3	10.6
8.50	1063	13.4	16.62	81	2.13	12.6	10.8
8.65	1064	13.7	16.88	81	2.14	12.9	11.1
8.80	1065	14.08	17.14	82	2.16	13.2	11.4
8.90	1066	14.4	17.40	83	2.18	13.5	11.6
9.00	1067	14.7	17.66	83	2.19	13.8	11.8
9.15	1068	15.0	17.92	84	2.20	14.0	12.1
9.25	1069	15.3	18.18	84	2.21	14.3	12.3
9.40	1070	15.6	18.45	85	2.23	14.6	12.5
9.55	1071	15.9	18.71	85	2.24	14.8	12.8
9.65	1072	16.2	18.97	85	2.25	15.1	13.0
9.80	1073	16.55	19.23	86	2.27	15.4	13.3
9.90	1074	16.9	19.49	87	2.28	15.7	13.5
10.05	1075	17.2	19.75	87	2.29	16.0	13.8
10.15	1076	17.5	20.01	87	2.30	16.3	14.0
10.25	1077	17.8	20.27	88	2.31	16.5	14.2
10.40	1078	18.1	20.53	88	2.32	16.8	14.4
10.55	1079	18.4	20.79	88	2.33	17.1	14.7
10.65	1080	18.75	21.06	89	2.34	17.4	14.9
10.80	1081	19.1	21.32	89	2.35	17.7	15.2
10.90	1082	19.4	21.58	90	2.36	17.9	15.4
11.05	1083	19.7	21.84	90	2.37	18.2	15.6
11.15	1084	20.0	22.10	90	2.38	18.45	15.9
11.25	1085	20.3	22.36	91	2.39	18.7	16.1
11.40	1086	20.6	22.62	91	2.40	19.0	16.3
11.55	1087	20.9	22.88	91	2.40	19.2	16.5
11.65	1088	21.25	23.14	92	2.41	19.5	16.8
11.75	1089	21.6	23.40	92	2.42	19.8	17.0
11.90	1090	21.9	23.67	93	2.43	20.1	17.3
12.05	1091	22.2	23.93	93	2.44	20.3	17.5
12.15	1092	22.5	24.19	93	2.45	20.6	17.7
12.25	1093	22.8	24.45	93	2.45	20.9	17.9
12.35	1094	23.1	24.71	94	2.46	21.1	18.2
12.50	1095	23.45	24.98	94	2.47	21.4	18.5

<sup>1</sup> See explanation of Table V. on p. 10; e.g., 1.068 sp. gr. = 5.3° Baume degrees, and  $5.3 \times 1.94 = 10.3$  of sugar. Results in col.  $e = \frac{c \times 100}{d}$ ; results in col.  $f = \frac{d \times 10}{a}$ ; results in col.  $g = \frac{c \times 1000}{b}$ .

## CHAPTER X.

### DEFECATION AND EVAPORATION OF CANE JUICE.

*THE cane juice and its properties.*—The cane juice as it comes from the mills is a straw-yellow opaque liquid—often of a dull grey or olive-green tint—which decomposes very rapidly, assuming a blackish hue. Its taste is balmy and sweet. It exhales the balsamic odour of the cane. The density varies from 1·033 to 1·106, according to circumstances. The juice, in fact, varies with the nature of the soil, with the season, the variety of the plant, and the manuring, whether extensive or the reverse. The fresh and more or less turbid juice may be separated into two portions—a liquid and a solid. The suspended solid portion may be separated from the liquid in which it is suspended by filtration or settling. The solid ingredients of the pressed juice consist of the cellular parenchyma of the cane, its woody fibres and bark, mixed with a green colouring principle similar to, if not identical with, chlorophyll. 100 parts of cane yield 98 to 110 parts of juice, according to the skill of the manufacturer and the quality of the cane.

*The juice requires speedy clarifying treatment on account of its liability to the acetous fermentation.*—In the tropics the expressed juice must be at once clarified; left to itself it is spontaneously liable to the acetous fermentation. Twenty minutes even often suffice to induce this destructive fermentative action.

*The clarified liquid still subject to the alcoholic fermentation.*—Even when the liquid has been clarified by the removal of its green fecula and albuminous matter, it is still liable to ferment. But the nature of the fermentation is now different; instead of an *acetous* the liquor is now liable to an *alcoholic* fermentation. Only such clarifying processes, therefore, as act in the desired manner as promptly as possible should be adopted.

*This fermentation formerly mistaken for a natural purification process.*—Now the West Indian sugar factory managers, seeing this blackish liquid rise rapidly to the surface in the form of a scum, carrying with it all the impurities of the juice to the top of the liquid, were accustomed to let it form, believing that it constituted



a natural purification of the *juice*, whilst all the time it was fermentation that had started. This fermentation was accompanied by a considerable formation of glucose at the expense of the sugar. The custom was thus a deplorable one. In cane-sugar factories, owing to the complete ignorance of the manufacturers, reforms are necessary at every step. It is not long since chemists were introduced into these torrid countries where mediocrity reigns supreme over the complete industrial vacuity of the natives, and it is only after extraordinary exertions that they have been able to obtain a footing by fighting against routine.

*Improved results from rational working.*—These efforts have already produced remarkable results. On those plantations where chemists have been located the yields have considerably increased, but in spite of such examples very few planters follow suit.

*Neutralising and boiling the juice.*—The juice as it comes from the mill should therefore be treated forthwith. As all sugar ferments are destroyed at 100° C. (212° F.), all destruction of sugar by ferments is avoided.

*The effect of the natural acidity on the sugar during boiling.*—But cane juice is naturally acid, and if an acid saccharine liquid be heated the sugar is *inverted*. It is therefore necessary to both boil and neutralise it at one and the same time. This process is what is termed *defecation*, this boiling having further functions still.

*Boiling and liming.*—The juice to be defecated is run into a jacketed pan, called the *defecation* pan. Steam is immediately run into the jacket. The liquid heats, and when it reaches about 80° C. (176° F.), from 2 to 3 lb. of lime per 100 gallons of juice say 2 to 3 grammes per litre, in the condition of milk of lime, are added and heat continued to be applied.

*The albuminous and waxy principles present in the juice.*—The cane juice contains albumen in abundance, and also other impurities which come from the pressure in the mill of the bark mixed with the vesicular tissue, such as the cerosin, a sort of waxy substance which it is necessary to separate. Cerosin is described on p. 238, and illustrated in Fig. 78. The liquid being saturated by lime and brought to the boil, a scum soon forms on the surface. It is the coagulated albumen which brings in its train (1) the excess of lime, (2) the cerosin, (3) the suspended matter, (4) in fact, all the substances which it is necessary to eliminate. There is thus formed an abundant scum, which is removed by skimmers. But heat must not be applied too long, because the pan would prime and overflow completely. A gutter is even arranged all around the pan to collect any froth or foam which may prime over.

*Fractional separation of the defecated liquor.*—When the steam is turned off the whole is allowed to settle. If the liquid be now sampled at different depths it will be found that the centre is quite

limpid, whilst the bottom and the surface are charged with suspended matter. The whole of the clear liquid is allowed to flow away through a tap with holes at different heights, then the bottom and top portions are run into tanks to be passed to the filter presses.

*Cane juice purification less complex than that of beets.*—The clear liquids, as well as that which flows from the filter presses, are now fit and ready for evaporation. It will be seen how simple it is to treat cane juice. It evolves no carbonatation or excess of lime. *Simple boiling after neutralisation by lime is all that is usually required in the purification of cane juice.*

*The juice from the sugar-cane diffusers still more easily treated.*—Sugar-cane diffusion juice is much easier still to work, because it is almost fit to evaporate just as it comes from the diffusers. Therefore they dispense with the defecation in some instances, if the juice has been treated in the diffusers themselves by a defecating agent, p. 265. In any case, a slight ebullition with a trace of lime suffices.

*The former dark-coloured juices entailed the use of bulky bone-black filters.*—When the cane juices were so badly wrought that they were full of glucose and colouring principles, energetic filtration through animal charcoal was necessary in order to get them to crystallise. At that time the sugar factories contained numerous charcoal filters, and the planters took enormous pride in showing the great extent of the filtration equipment of their factories.

*Bone-black filters at the present day a sure sign of bad working.*—At the present time it is all the other way about. A sugar factory containing a great number of bone-black filters is a badly conducted one, because modern equipped sugar factories do not now use bone-black filters, which are now confined to the refinery.

*The suspended matters the chief impurity to be eliminated.*—Everywhere mechanical filters and occasionally sand filters are to be seen; the juice as it comes from the mill or the diffusion—that coming from the defecation pans or the filter presses—are all filtered through bagasse or cloth or sand to free them from suspended matters, the real enemies of good working.

*Cambray's process for the rational purification of sugar-cane juice.*—Although from a mechanical point of view the great progress made in the sugar-beet industry has extended itself to the manufacture of sugar from the sugar cane, yet this is not the case in regard to the yield of sugar obtained, owing to the want of a rational process for purifying the cane juice. The latter contains, besides sugar, variable quantities of glucose, gummy substances, etc., which exert a great retarding influence on crystallisation of the sugar and induce rapid fermentation of the latter. That is why the glucose increases throughout the different stages of manufacture at the expense of

sucrose. The object of Cambray's process, therefore, is to eliminate the gums and the principal mineral and organic impurities, thus reducing the glucose present and avoiding any future formation thereof. The yield is increased and the final quantity of molasses reduced to a minimum. It consists in the double sulphitation of the cane juice in the presence of lime; from  $3\frac{1}{2}$  to 4 lb. of quicklime, in the form of milk of lime, is added to every 100 gallons of the juice as it comes from the mills or the diffusers, the temperature of the juice being kept between  $50^{\circ}$  to  $55^{\circ}$  C. ( $122^{\circ}$  to  $131^{\circ}$  F.). A current of anhydrous gaseous sulphurous acid, produced by a special apparatus (Fig. 92), is then passed through the liquid, and the sulphitation continued till the filtrate gives an alkalinity corresponding to 0.2 to

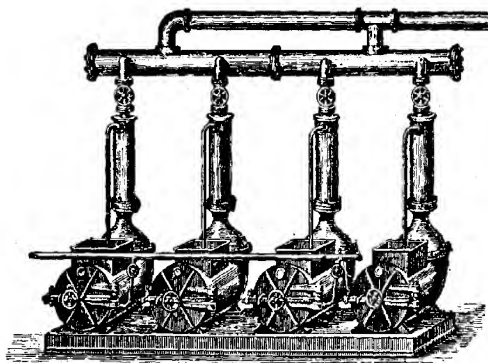


FIG. 92.—Sulphur kiln battery (CAMBRAY).

0.3 gramme of lime per litre; the turbid juice is then run into decanting tanks. The muddy matters settle quickly, and the juice may be readily decanted. The sludge is filter pressed, and the clear juice brought to the boil and again treated with sulphurous acid until slightly acid. The sulphited juice is not only much purer than the crude juice, containing less glucose, etc., but it is much less liable to fermentation than the latter, thus facilitating subsequent operations. It is very easily evaporated and boiled down, and four successive crystallisations may be obtained from it, the sugar being of high quality. The following results were obtained in a cane-sugar factory which used this double sulphitation process in 1895, the ordinary processes having been previously followed:—

# DEFECATION AND EVAPORATION OF CANE JUICE 279

		1894.	1895.
Purity	{ Diffusion juice purity	77.86	82.96
	{ Syrup purity	76.60	84.62
	Difference	- 1.26	+ 1.66
Non-saccharine.	{ Non-saccharine, diffusion juice	27.79	20.52
	{ Non-saccharine, syrup	30.04	17.95
	Difference	+ 2.25	- 2.57
Glucose per cent. of sugar	{ Diffusion juice	18.48	7.17
	{ Syrup	14.53	4.89
	Difference	+ 1.05	- 2.28

These figures speak for themselves. Identical results have been observed in the composition of the products of later stages :—

		1894.	1895.	Difference.
Seconds—Purity		48.19	71.21	+23.02
" Glucose per cent. of sugar		51.80	12.51	-39.29
" Non-saccharine per cent. of sugar		103.26	40.54	-62.72
Thirds—Purity		35.27	56.18	+20.91
" Glucose per cent. of sugar		91.93	25.87	-66.06
" Non-saccharine per cent. of sugar		183.30	78.27	-105.03

The average analyses of the *masses cuites* of the first and second sugars from this process are as follows :—

	1st Masse Cuite.	2nd Masse Cuite.
Degrees Baumé	48.16	47.6
" Brix	92.7	91.2
Sugar	78.92	61.8
Water	10.05	13.53
Glucose	3.99	8.5
Ash	3.28	7.25
Organic matter	3.73	6.92
Purity	87.77	73.16
Glucose per cent. of sugar	5.05	13.75
Saline coefficient	24.06	8.52

*Sulphitation pan.*—This pan (Fig. 93) is made of very strong wrought iron, with the bottom sloping towards the discharge valve so as to ensure as complete evacuation as possible. It is furnished with a steam coil, a steam valve, a juice tap, a gauge tap, and a discharge tap, wrought by a wheel attached to a rod, which is actuated on from the upper part of the pan. It can treat a layer of liquid a metre in depth or more.

*Sulphitation of the syrup.*—Much sulphurous acid is used in cane-sugar factories as a decoloriser and defecating agent for the syrups. This sulphitation produces very good effects both on the colour and the yield of sugar, see p. 297.

*Evaporation.*—The old method of boiling down the juice in a row of open pans set over a furnace and its flue.—The defecated juices have (1) to be evaporated and (2) boiled down to crystallising-point. Although there is no necessity to dwell on the subject, we must here say a few words about the ancient naked-fire methods which still subsist in the

old factories. The process has been described in the older manuals until the descriptions are about as stale as the process itself is wasteful. The juices are run into a series of pans placed in single file on the same furnace. The first, the largest pan, on that account is called the *grand*, is immediately above the fire. The others, which gradually become smaller, are to the rear; and the last of all, the smallest, is next to the chimney, where the flame is weakest. The juice is run into No. I., No. II. is charged from No. I., No. III. from No. II., No. IV. from No. III. The juice thus becomes denser and denser until it reaches the last, when it is in the condition of syrup.

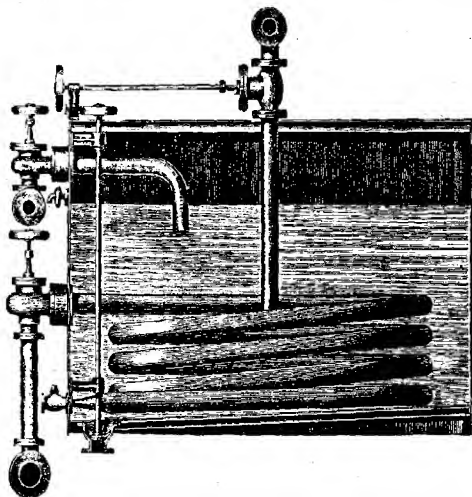


FIG. 93.—Sulphitation pan (CAMBRAY).

*Claying.*—Formerly this syrup, when well concentrated, was poured into sugar-loaf moulds, then carried into the crystallisation-room, where it solidified on cooling. This mass was then *clayed*, i.e., its surface was covered by moistened clay. This moisture percolated through the loaf to its apex, driving the molasses before it. When the mould was undone, the paste, covered with clay, was removed, the apex full of molasses was detached and a more or less yellow loaf was left, which represented the purest product of the manufacture.

*Concentrating the juice in open-air pans heated by steam.*—Later on

evaporation was effected by steam in the open air. The most improved appliances were Wetzell's pan and Chenailler's pan. In the middle of a trough filled with cane juice a hollow axis revolved, furnished throughout its entire length by hollowed-out enlargements in the form of lenses. Steam circulated in the axis, and filled the lenses, which came half out of the liquid. The juice moistening the lenses, being unceasingly renewed by the rotation and by some receptacles fixed on their exterior which were discharged from above, was in the form of this thin layer evaporated very quickly, and soon was converted into syrup fit for boiling. Then boiling was done in Howard's vacuum pans, the first which were wrought in a vacuum. At the present time evaporation is effected by means of the triple and quadruple-effect, as in beet-sugar works, and *the remainder of the work in no way differs from that already described: boiling to strike-point, turbinng, etc.*, as in the case of beets. Minute details would mean repetition.

*Molasses.*—The final cane-sugar molasses are sold to the distilleries for the manufacture of rum or for general consumption. Cane-sugar molasses have, in fact, a perfumed taste which is very agreeable, the opposite being the case with beet molasses. The natives of sugar-cane countries, and even the British and sailors in general of all countries, consume enormous quantities of it. *Molascuit.*—Under this somewhat misleading trade name a mixture of bagasse and molasses is exported to Europe for cattle feeding. As a cheap method of drying molasses for transit it may have some merit, but in any case it deteriorates their own excellent feeding value *pro rata* with its undigestible fibre-content. It is to be hoped the practice of dumping molasses into the sea, as is done in the French Colonies, will cease, and they will be converted into industrial alcohol—a promising source of motive power.

The second quality of cane sugar is yellow and has a delicious taste. It is accordingly consumed directly in this condition, and many prefer it to the first quality white sugar, in which this perfume is much less pronounced. But genuine "Demerara" is rare, whilst dyed imitations, often from poisonous dyes or reagents, abound.

TABLE LIV.—SHOWING THE MEAN COMPOSITION OF FOURTH MASSES CUTES AND MOLASSES THEREFROM AT MAGNOLIA, 1888-89.

	Sucrose direct polarisation.	Sucrose double polarisation.	Glucose.	Coefficient of purity (direct polarisation).	Coefficient of purity (double polarisation).
	Per Cent.	Per Cent.	Per Cent.		
Molasses	80.00	38.98	17.6	37.93	42.68
<i>Masses Cutes</i>	44.45	48.79	17.45	...	...

Such are the points to be noted in the manufacture of sugar from the cane, only the first stages of which differ from that of the beet. All the other processes are identical; the analyses are the same. All the sugar that is not consumed on the spot is bought by the refiners. The cane-sugar market is therefore the same as the beet-

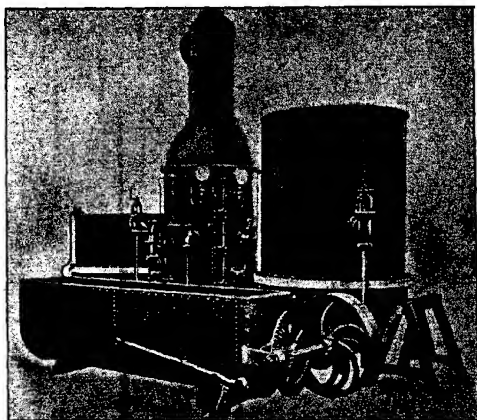


FIG. 94.—Compact cane sugar plant for colonial homestead (CAIL).

sugar market. They thus follow the same quotations and the same fluctuations of the market. The chemistry of molasses is dealt with on pp. 395 *et seq.*

Fig. 94 shows a compact sugar-cane plant, suitable for the colonies, consisting of vacuum pan, defecation pan, cooler, etc.

## SECTION III.

### CHAPTER XI.

#### SUGAR REFINING.

*THE raw material.*—Most refiners, when at all practicable, buy their sugars in one out of three possible conditions only: (1) White sugar. (2) Yellow sugar, with 88 per cent. yield. (3) Brown impure sugar. Some refiners buy sugar under one of these conditions only, *e.g.*, Russian refiners only treat white sugars, whilst certain Hamburg refineries only treat the black impure sugar from the Réunion. Again, the Clyde refiners, in their day, only bought sugar with less than 4 or 5 per cent. of fruit sugar.

*The purchase and selection of the raw sugar.*—Several points require attention in selecting the raw material. The quality selected will, to a great extent, depend upon the method of refining in vogue in the purchasing refinery. There are many varieties and qualities of raw sugar, each of which lends itself more or less to some particular method of refining. The bright yellow sorts are almost exclusively monopolised by grocers for sale in original muscovado condition. Hence, darker and duller tinted varieties are only available for refining. In some refining systems, *e.g.*, those in vogue in Greenock, when the sugar-refining industry attained its zenith in that town, no syrup was made. It was then absolutely essential to use sugars which contained only a comparatively small amount of uncrystallisable sugar—say, not exceeding, on an average, more than 3 or 4 per cent. But where a considerable quantity of syrup is turned out, the quality of the sugar is not so important a matter, and low-quality sugars such as *Syrup Mauritiæ*, *Jaggery* and *Manilla*, may be used (Wallace). Again, judicious mixtures may often be made, such as beet sugar with sorts rich in fruit sugar; but where no syrup is made, as in the Greenock system, too large a proportion of



beet was guarded against, as the soluble saline matter of the beet sugar of those days had a tendency to become concentrated in the lower qualities of the refined article, not only hindering its crystallisation, but imparting to it a peculiarly nasty taste.

TABLE LV.—ANALYSES OF RAW SUGAR.

	Cuba Muscovado.	Grenada.	Trinidad.	Demerara.	St. Vincent.	Concrete.	Melado.	Molasses.	Pernambuco.	Paraita.
Cane sugar . .	92.35	92.31	90.41	90.80	89.00	84.20	67.00	47.0	88.81	84.00
Fruit sugar . .	3.38	4.06	3.84	4.11	5.85	8.45	11.36	20.4	4.82	6.00
Extractive, etc.	.06	.66	.95	.77	.76	1.70	1.93	2.7	.94	1.28
Soluble salts . .	.02	.37	.86	.92	.62	1.10	.76	2.6	.80	1.20
Insoluble . . .	.15	.94	.22	.20	.05	...	.15	...	.73	1.10
Water . . . .	2.84	2.56	3.72	3.20	3.72	4.65	18.80	27.3	4.40	5.52
	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00
Colour, D.S. .	13	13	10	10	10	12	...	...	8	8
Cane sugar obtainable .	85.9	86.4	82.8	82.1	80	70.2	51.8	13.6	79.5	72.9
	Bahia.	Syrup Mauritius.	Calcutta Date.	Jaggery.	Unclayed Manilla.	Amoy.	Formosa.	Maple.	Fr. Beet (fine).	Ger. Beet (low).
Cane sugar . .	86.80	86.00	87.06	86.73	79.00	74.60	76.53	72.60	94.30	87.80
Fruit sugar . .	5.03	6.35	6.95	6.05	11.76	16.13	13.38	13.95	.25	.33
Extractive, etc.	1.72	1.62	.65	1.29	1.32	1.70	2.47	2.11	.27	.75
Soluble salts . .	1.21	1.44	.63	.88	1.95	1.61	1.86	1.35	1.30	5.92
Insoluble . . .	.92	.23	.56	2.01	.63	.54	.16	.03	...	.13
Water . . . .	4.32	4.36	4.10	3.04	5.34	5.52	5.60	9.96	3.83	5.07
	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00
Colour, D. S. .	7	8	8	6	6	7	9	...	13	10
Cane sugar obtainable .	75.8	72.5	76.7	76.3	57.5	50.4	53.8	51.9	87.5	57.9

The total amount of cane sugar in any given lot of the raw material is of prime importance. On it depends the quantity of refined sugar which it is possible to produce from the raw sugar in question. Next in importance is the amount of fruit sugar as well as

of soluble saline matter (soluble salts), both of which to a great extent retard or prevent the crystallisation of the cane sugar. The amount and nature of the extractive matter containing the colouring principles determine the amount of animal charcoal; whether greater or less, which it is necessary to use in the refining process, as well as the extent to which, and the manner in which, the charcoal will thereby be deteriorated. The amount and nature of the insoluble substances in the raw sugar is also an important point to be considered. When present to any great extent, difficulty is experienced in washing. Moreover, in some cases it is exceedingly troublesome to filter—the coarse particles, on the one hand, choking up the filter cloth, whilst the finer particles pass through the filter cloth, to be intercepted by the animal charcoal, the decolorising properties and other functions of which it greatly deteriorates. Some varieties of East Indian sugar are peculiarly subject to this defect. Each refiner strives *never* to have to work except upon one and the same species of sugar. The work is therefore arranged accordingly, and is never altered, so that the workmen know what they have to do, and could do it blindfolded. They are thus always sure of bringing their refined sugar to the same percentage by following their instructions. Refiners should therefore regulate their work according to a product of predetermined quality, so that the routine of this work should always be the same and no error creep into the manipulations; for there is nothing more dangerous in an establishment comprising a large number of workmen than to keep incessantly changing the method of working. If it does not give rise to mistakes, there is inevitable vexation, loss of time and money. But it is only possible, in the case of small establishments, to always treat the same material, except in Russia, where the refiner, being the only buyer of native sugar, can lay down the law to the factories and require them not to deliver any but extra-white sugar. The large sugar refineries of Paris, which work up enormous quantities of sugar, cannot choose and pick, and are forced to take all as they come. They must so regulate the quality of the sugar which they send to the melting pans that the subsequent operations may always be the same. The refineries therefore consist of two distinct factories or departments: (1) That in which the sugars are sampled and blended so as to make mixings of a predetermined yield from them; and (2) the refinery, properly so called, where those mixtures are melted and undergo further treatment.

*Blending sugars prior to refining.*—Sugars are bought in immense quantities and piled in vast warehouses, where they are classified—cane sugar on the one hand, beet sugar on the other. All these sugars are analysed so as to determine their percentage of sugar, ash, alkalinity or acidity.

*Blending the alkaline beet sugar with the acid cane sugar to produce*

*a neutral mixture.*—Beet sugar is alkaline, cane sugar is acid. A neutral sugar is got by mixing cane and beet sugar in the right proportions.

Again, if there be in stock white sugar and brown sugar, and if the work of refining be regulated on the treatment of sugar as titrating 88°, the white and brown sugars are mixed in the requisite proportions to produce 88°. All this requires a profound knowledge of the nature of sugars, their origin, and so on; but it is a question of every man to his trade, and the expert is trained both by science and practice.

*Saline impurities.*—*Preliminary turbinizing.*—However, things are not quite so simple as all that. It is always desirable to remelt as pure sugars as possible. The most dangerous impurities are the saline matters, and these are present in the molasses which soil the sugars. If the raw sugars were freed from these molasses, products would be obtained ready for treatment containing much less salts, and the yield on refining would be greater. The first treatment therefore to which brown sugars of a certain grade are subjected is to free them by turbinizing them from the molasses which they contain. Therefore the first department met with in a sugar refinery is a vast installation of turbines, the extent of which is of greater and greater dimensions, according as the sugars are brought to a greater and greater degree of purity. The raw sugar is first mixed, sieved if its nature will allow, introduced into the turbines, equalising the charge as much as possible, then clarified by a blast of steam (p. 212), until the desired tint has been obtained by the expulsion of the molasses which discoloured the crystals. Some refineries make quite an industry of this turbinizing. Certain "seconds" clarified with sugar liquid become quite white when turbinized. Then when well sifted and mixed with ultramarine blue, and turbinized under those conditions for a long enough time for the crystals to amalgamate together, the cakes are taken from the drum, crushed and delivered for consumption as *granulated*. This special refining is cheap and profitable, because the sugar which it produces has the same properties as refined. Moreover, the molasses which it produces yield, on reboiling, sugars which are melted and refined in the ordinary way. In Italy, where granulated sugars are easily sold, several large establishments work thus, with so much more profit, because, the duty on sugars being paid according to tint, it is easy to procure very rich highly coloured sugars which turbine very well. All countries which pay the sugar duty by the tint are similarly situated. In French factories the turbinized sugars, brought to the desired strength, are mixed. The sugar is shot from the sacks into a larger hopper in the right proportions, white, brown, beet or cane, to obtain the desired type. All these are mixed and melted. This preparatory work is the only one that requires attention from the

refinery manager, who, aided by the laboratory and his special knowledge of sugars of different origin, ought to always maintain a constant supervision on all the operations which occur in this first stage of the process.

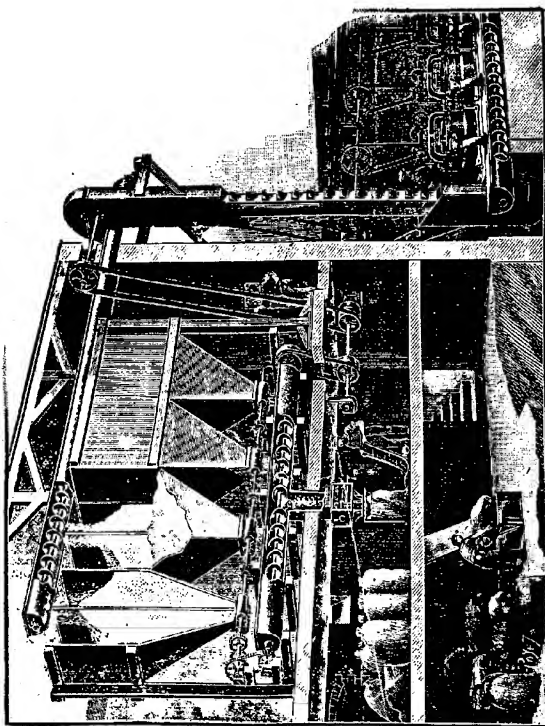


FIG. 95.—Battery of French turbines with automatic conveyor of turbinéd sugar to mixer and bagging-up machine.

*Solution or "blowing-up".*—But where the special initial turbine is not in vogue, the first stage in the sugar-refining process consists in dissolving the raw sugar in water, so as to obtain a thick solution or syrup. The effect of different qualities of water—hard, soft, etc.—on the char will be adverted to further on.

*Open-fire system of heating in early days.*—In the early days of the sugar industry this was effected by heating the sugar and water in the right proportions over an open fire—a method long since abandoned, a steam heat being adopted instead.

*Steam heat.—Its effect on sugar solutions.*—By the use of steam as a source of heat, all risk of burning the sugar is avoided. Steam heat, however, is not without an injurious action on sugar, similar to that of burning by direct heat. The extent of this injurious action depends principally on the pressure of the steam used; that is to say, if it be used to heat the saccharine solution in the form of open or free steam, that is, by being injected into the liquid through an open pipe. Two methods of applying steam for dissolving sugar have been successively in vogue :—

*Naked steam.*—Formerly the sugar and water were exposed to the action of open steam, that is, to the action of naked jets of steam injected into it from a series of perforated tubes of iron or copper, ramifying below a false bottom placed across a rectangular metal tank. Owing to the violent agitation and deafening blowing noise produced by the condensation and escaping of the steam, the name of "*blowing-up pan*" was applied to the tank in which the process was conducted, and the appropriate term of "*blowing-up*" was applied to the process itself. The action of the open steam dissolves the sugar very rapidly, the steam jet not only supplying the source of heat, but also the means of so agitating the liquid as to keep it in continual motion, little or no stirring being required.

*Applying heat by means of steam jackets and close steam coils.*—It was soon, however, found in actual practice, that, when naked steam was brought into contact with a solution of sugar in the manner described, it is much more injurious than when the solution, contained in a suitable vessel, is heated by applying steam to the outside of the vessel, *e.g.*, as in what is known as a steam-jacketed pan. The old-fashioned blow-up pans therefore were eventually done away with, in favour of vessels constructed on the steam-jacket principle, or of vessels heated by means of a close steam coil (Fig. 101, A).

In sugar refining as it was practised extensively in Greenock, the different floors of a sugar house, generally six or seven, were arranged so that in the process the sugar passed down from one to the other. The sugar was accordingly hoisted to the top story, where it was emptied out of the hogsheads, boxes, baskets or bags in which it had been imported, and then shovelled through holes in the floor into the blow-up pans, which are situated in the floor underneath. The blow-up pans are cast-iron vessels, some four or five feet deep by six to ten feet in diameter. A pan of ten feet deep diameter will dissolve in the necessary quantity of water about eight or ten tons of raw sugar. At a little distance from the real bottom of the pan is a false bottom perforated with holes. The

sugar rests on this false bottom until dissolved. Between the false and the real bottom a series of steam pipes are laid. The false bottom prevents the solid sugar coming in that condition in direct contact with the steam pipes, and being partially burnt or decolorised. A mechanical agitator, fitted with blades, is made to revolve by suitable gearing both above and below the false bottom, so as to keep the liquid continually in movement. The operation is commenced by running into the pan a sufficiency of water, or thin liquor. Here, it may be remarked that the dissolving of the sugar in the upper floor of a refinery, owing to the great height, is more practicable in localities where a good natural supply and pressure of water exists. Where there is but a feeble pressure of water, it may be better to dissolve the sugar on the ground floor and pump it up by means of an "air vessel" or "egg" or *montejus* to the top floor, to undergo the other stages of refining in its descent.

In any case, the water or thin liquor having been run into the pan in sufficient quantity, the steam is turned on to the steam coil, and the sugar shovelled in as fast as it can be emptied out of the casks. The filling of the pan takes about half an hour, and the liquid should by that time have attained a density of 28° Baumé (45° Twaddell, sp. gr. 1.225), and the temperature as near as may be 180° F. (82° C.). There are several varieties of Baumé's hydrometers, each variety with its own special graduation, hence much confusion. The actual gravity and degree Baumé given above are both quoted from Wallace. The point to be noted is that *there is two of sugar to one of water, and if pure sugar were nrought with, that would give a saturated solution at 15° C.*, what is very evidently aimed at. Such a saturated solution has a density of 1.326 at that temperature, and would approach at 82° C. that given by Wallace very closely. The liquid then contains *about* two parts of raw sugar to one of water. As regards the density or specific gravity of the liquid, there may be considerable variation. In fact, the operation of effecting solution—or "blowing-up"—is not applied to *carefully* weighed or measured portions of sugar and water, but so much of each are run in as the operator may deem necessary to get the desired strength, his guide throughout being the saccharimeter of Baumé. The sugar to be employed is scattered loosely over the floor, and the workmen throw it into the solution tank like so much earth. No two refiners are exactly agreed as to the exact density. Perhaps that which we have just given, due to Wallace, may be the best. Horsin-Deon gives 37° to 39° B. (sp. gr. 1.34 to 1.36 at 17°·5 C.). Hitherto only a solution of raw sugar in water has been dealt with, and during the heating of this solution a scum rises to the surface, which is skimmed off by almost flat perforated ladles, but the amount of flocculent insoluble material skimmed off in this way is really very trifling. But be that as it may, this is the simple manner

in which the solution was effected in most of the old Clyde refineries. In other localities, however, the liquid is subjected to different kinds of treatment in the "*blow-up*," so as either to remove a portion of the colouring matter or to neutralise any traces of acid in the sugar. This acid may be either pre-existent in the sugar (malic acid, etc., p. 343), or be developed during the stay of the sugar in the hold of the ship, or in the warehouse (acetic acid, lactic acid). Again, the liquid may be treated in the pan with a view to facilitate subsequent filtration, so as to produce a perfectly clear and bright liquid. To neutralise acidity when working with cane sugar alone without any admixture with beet, it is usual to add enough milk of lime or lime water. Although not general even in some of the most skilfully managed refineries, it may yet be regarded as an advantageous practice in many instances. But it must not be used in excess, otherwise it will darken the liquor and thus throw extra work on the char filters. The lime is said to be employed for the purpose of correcting acidity, but this explanation is not altogether correct. It is not complete. If only mere neutralisation of acidity were required, *i.e.*, if we use the term acidity in its ordinary acceptation, chalk should succeed as well as lime, and its use would be free from many of the risks of injury to the sugar with which the use of lime is attended. Again, it has been suggested that the existence of an acid in unfermented sugar is hypothetical, and, moreover, if the antacid quality of lime were especially desired, lime water, containing as it does so little of that earth, would be a very inefficient medium of administration. The real function of lime water in sugar refining would appear to be that its lime combines with the various colouring and gummy principles existing in raw sugar to produce a flocculent precipitable substance. Again, bullock's blood, in virtue of its coagulation by heat, was formerly added to the blow-up pan, and was technically known as *spice*. When the bullock's blood coagulated, it enveloped, in the albumen of the serum, the flocculent precipitable substance produced by the action of the lime on the colouring matter of the sugar, as well as any other impurities present; and the whole floating to the top of the liquid in the form of easily removed scum, the sugar liquor was more or less clarified. The coagulated albumen, in virtue of its affinity for colouring principles, was said to exert of itself a bleaching action on the solution of sugar. As more cleanly substitutes for blood, solid albumen obtained by appropriately evaporating white of eggs, or the serum of blood at a low temperature, has been used. But these substances render the syrups very impure, and are now but seldom used. Horsin-Deon, however, still mentions  $1\frac{1}{2}$  to 2 per cent. of ox blood, and 2 to 4 per cent. of fine char, as well as sometimes a little caustic baryta, to precipitate certain organic and mineral matters. Amongst the various substances suggested as substitutes for blood, a mixture

of sulphate of alumina and superphosphate of lime, the ingredients of which react on each other to form a gelatinous precipitate, may be mentioned; but this mixture is objectionable, because it introduces calcic sulphate, soluble phosphate of lime, and soluble phosphates of alumina and lime. Spencer, in his report on the diffusion *versus* milling work at Magnolia, U.S.A., says: "I have frequently objected to the use of superphosphate of lime and alumina. These superphosphates usually contain an excess of the sulphuric acid used in their manufacture, and the stock at Magnolia was no exception to the rule. It is difficult to find any advantage arising from the use of these superphosphates." Again, superphosphates are objectionable as made from acid itself made from arsenical pyrites. According to Wallace, the greatest objection to these mixtures is the danger of the common workmen, who are necessarily entrusted with the application of them, using excess of one or another and thus rendering the liquor acid or alkaline, and so doing more harm than good—more especially as it would not appear to be indispensably necessary to add anything to the liquor, as many refiners of the first rank use no chemical reagent. Animal charcoal dust is sometimes introduced into the blow-up pan, so as to decolorise, or partially decolorise, the sugar solution. Provided the charcoal be new, that is, has not been used before in sugar refining, it produces a good result; but, owing to its getting contaminated with the impurities in the sugar, it can only be used once. Char dust is many times more powerful than granulated char. An abundant scum is formed, which is continuously broken by the agitator. The whole is then passed through the Taylor filters (Figs. 96 and 101). Sometimes clarification is effected in other tanks (Fig. 101, L) from those in which the sugar is melted. When the mixture of syrup, blood, black and baryta is effected, this liquid is pumped by a *montejus* (Fig. 101, H) or a pump into a closed pan (Fig. 101, L), where a slight vacuum is made which induces boiling and agitates the mixture. The vacuum is broken by and by, the temperature is raised to 105° C. so as to complete the coagulation of the blood albumen, after which it is allowed to flow into the bag filters (Fig. 101, N). Filtration, therefore, involves two operations—(A) passage through Taylor's filter bags; (B) through bone char.

*A.—Removal of suspended impurities by bag filtration.—Taylor's filters.*—The clarified syrup must be freed from any suspended matter—coagulated blood, black, and precipitates of all kinds. It is therefore filtered through Taylor's filters (Figs. 96 and 101). These filters are of two kinds; either the liquid enters by the inside, depositing its impurities within the bags—or the bags, kept stretched by an inside lining of osiers and packed in tight cases, dip into the liquid, which passes through them, leaving all the impurities behind in the case. No matter which of the two methods be adopted, the filtered



syrup should be quite limpid. When the mixture of sugar and water has become thoroughly dissolved, and is of the appropriate density, and when the coarser impurities which have risen to the top of the liquid in the form of scum have been skimmed off, a sluice or tap M is opened in the bottom of the pan (Fig. 101), and the fluid is allowed to run into a shallow tank or tray (Fig. 96, *a* and Fig. 101, N), to the bottom of which a very ingenious system of filtration is adjusted. In the bottom of the shallow tank *a* (Fig. 96) there are numerous apertures *o, o, o, o*, each of which is cut with a screw into the form of a female screw. Into each of these a brass filter bell *b*, terminating in a tube in the form of a male screw, is tightly screwed. To each of the filter bells there is securely tied at *d* a filter bag *c*, of twilled cotton, in the shape of a pillow-case or bolster, of about two feet in diameter, and each of these bags is thrust into another bag *s*, of coarse canvas of about six inches in diameter. The outside bag is very appropriately termed a sheath. These bags are some six or eight feet long, and are fastened, as described, to the number of 200 or so to the bottom of the shallow tank in question; the whole being surrounded by the sides of an iron box (Fig. 101, N), so that the liquid may be kept hot by means of steam introduced for the purpose. It will thus be seen that this filtering arrangement is well adapted to provide a wide superficial area in a compartment of comparatively small cubic content—so necessary in a sugar refinery where operations are conducted on so large a scale in premises of not unlimited dimensions. The buildings would be spread over too wide a superficial area of ground, if space were not utilised to its fullest extent without cramping. The above system of filtration is therefore a very happy and effective illustration of the adjustment of means to an end. Not only is it economical in space, but it is about as effective a means as can be imagined of what might be called automatic filtration, in contradistinction to filtration through filter presses. Other mechanical filters are described on pp. 118-123.

*Points which require attention in bag filtration.*—Several points require attention during filtration, the most important is to fill the bags with the sugar liquor in as short a time as possible. Unless the bags are completely full the filtrate comes away foul. The filtering arrangement (Fig. 101, N) must be warmed up to the right temperature before the liquid is run off from the blow-up pan. This is done in a very simple but effective manner by suspending the filter bags in a box or chamber, into which steam jets may be turned on or turned off at will. The filtered liquid as it comes from the filter bags is sampled and examined from time to time by a workman. He takes a sample in a test-glass, and examines it by transmitted light to see whether it is perfectly bright. The brightness of the liquid is a very important point, as any turbidity injures the quality of the sugar-loaves to be made from it in the subsequent operations. Until

the filtered fluid comes away perfectly clear and bright it is run into a receiver called the foul-liquor tank, from which it passes back to the blow-up pan. It has been suggested to replace the Taylor filters by filter presses, which answer very well if the filter cloth has been well selected. But sometimes it is necessary to add solid



FIG. 96.—Taylor's filter bags.

matters in suspension to counteract the gluey nature of the muddy residues left on the filter cloths, which would otherwise choke up its pores. This sludge, called refinery black, forms an excellent manure, containing the phosphate in the black, the nitrogen of the blood, the sulphuric acid and salts, and the whole of the other organic matter.

This manure is deservedly held in high esteem by farmers *intelligent enough not to object to its black colour.*

*B.—Decolorising of the solution of raw sugar.—Removal of dissolved impurities.*—1. *By animal charcoal.*—When the maximum degree of brightness and freedom from turbidity have been obtained, if the filtered liquid be examined it will be found that, however bright and clear the liquid may be, its colour will be dark, not unlike that of port wine. The next process, therefore, after having got the solution clear and bright, is to get rid of the colouring matter—to decolorise it, in fact, or to bleach it. This is accomplished more or less effectually by passing it through or bringing it into contact with more or less finely ground bone black. This substance is the same as that often sold to artists under the misleading name of ivory black. The excellent decolorising properties of animal charcoal may very well be illustrated, or demonstrated, by agitating a small quantity of port wine, tincture of litmus, cochineal, or any other coloured solution with it. Or, better still, if the coloured solution be passed through a layer of bone black, the filtrate will be more or less completely decolorised.

*The use of other charcoals impracticable.*—Although wood and other charcoals also possess decolorising properties, animal charcoal has been found practically to be most suitable for the purposes of the sugar refiner. Indeed, many varieties of charcoal have been tried. Some of these are much more energetic and effective as decolorisers than bone black. But, owing to other and objectionable properties, it is not practicable to render this superiority in decolorising properties available in sugar refining.

*Peculiar adaptability of animal charcoal.*—Animal charcoal only is endowed with that essential aggregation and combination of characteristics and properties required in any substance which has to effectively fulfil the rôle of a decoloriser of sugar solutions. Such a substance must be free from soluble inorganic salts, that is, from salts which readily dissolve in water. Neither must it contain any very appreciable quantity of sulphate or carbonate of lime. Whatever decolorising agent may be used, its specific gravity must be at least so great that it will sink freely in the densest of sugar liquors, whilst at the same time it must be extremely porous; and not only so, but it must be hard and coherent enough to bear handling and the rough usage to which it is subjected in being carted and shovelled about and reburned every other fourth day for several years in succession. No charcoal fulfils these many and varied requirements in such a marked manner and to so great an extent as animal charcoal. By animal charcoal is meant the charcoal made from bones. Charcoal made from such substances as leather waste, blood, etc., might be regarded as animal charcoal. Yet, although leather charcoal is highly esteemed in the metallurgy of the noble

metals, for many reasons it is unsuitable for sugar refining. The practice of animal charcoal manufacturers and sugar refiners of quenching the red-hot charcoal as it comes from the kilns with water is greatly to be deprecated, as being highly injurious to the decolorising properties of the char. The water acting on the red-hot nitrogenous carbon evolves ammonia, and thus diminishes the proportion of the nitrogen, which is supposed to play such an important rôle. Some animal-charcoal makers strive, for the sake of a little extra profit, to distil off as much ammonia from the bones as possible, so as to secure a larger yield of sulphate of ammonia, and it has even been suggested to add lime and inject superheated steam into the retorts. No doubt such innovations would yield a larger quantity of sulphate of ammonia per ton of bones burned, but it would inevitably deteriorate the decolorising properties of the resulting charcoal, and in the case of lime spoil it altogether.

*Selection of good quality charcoal.*—This is a most important point. New animal charcoal—that is, freshly calcined bones—consists essentially of carbon, tribasic phosphate of lime ( $\text{Ca}_3\text{P}_2\text{O}_8$ ), and carbonate of lime ( $\text{CaCO}_3$ ), together with small proportions of a few other substances. Its composition is not definite, varying somewhat with the source of the bones. The following analyses by Wallace give some idea of its general composition:—

TABLE LVI.—ANALYSES OF NEW ANIMAL CHARCOAL.<sup>1</sup>

Dry.	A.	B.	C.
	Per Cent.	Per Cent.	Per Cent.
Carbon (nitrogenous) . . . . .	9.71	7.64	10.37
Calcic phosphate . . . . .	80.48	84.05	78.70
Calcic carbonate . . . . .	8.82	7.61	8.05
Calcic sulphate . . . . .	0.81	0.20	0.53
Alkaline salts . . . . .	0.30	0.25	0.58
Ferric oxide . . . . .	0.12	0.15	0.21
Silicious matters . . . . .	0.26	0.10	1.56
	100.00	100.00	100.00
Cubic feet per ton . . . . .	51	49	47

A, from ordinary bones. B, from South American shank bones. C, from camp bones frequently buried for some years before collection.

*Nitrogen, etc., in animal charcoal.*—As there is a very appreciable quantity of nitrogen and a small amount of hydrogen in the so-

<sup>1</sup> Analyses of old and used chars are given on p. 306.

called carbon of animal charcoal, it is far from being pure. The proportions of both these elementary bodies present in any given char depend upon the temperature to which the charcoal has been exposed in the charcoal burners. As a general rule, the nitrogen amounts to about one-tenth part of the total carbonaceous matter, but occasionally it is even more. Char which has been used over and over again in sugar refining and reburnt in the interval does not contain so much nitrogen, and the proportion would appear to diminish in a direct ratio with the number of times the charcoal has been reburned and re-used in sugar refining. In a not very old charcoal it was found to be as low as 0.3 per cent., and no doubt in still older charcoal would be much less. The amount of hydrogen in well-burnt animal charcoal is very small indeed. Dr. Wallace quotes a case in which a new char only contained 0.034 per cent.

*Function of the nitrogenous carbon in animal charcoal.*—The whole rationale of the decolorising effects of animal charcoal is shrouded in mystery, and no point is more obscure than the rôle of the nitrogenous carbon. That it plays an important part there can be no manner of doubt. Some authorities even go so far as to assert that it is an essential constituent of animal charcoal, and that no char which does not contain an appreciable quantity of nitrogen is a good decolorising agent. It has been pointed out that wood charcoal, for example, although exceedingly permeable and an excellent absorber of gases, is a very poor decolorising agent, and is practically useless for the purposes of the sugar refiner.

*The injurious action of the presence of free ammonia and of ammonium sulphide in new char.*—Traces of free ammonia are always present in new charcoal as originally received from the animal-charcoal maker. The proportion, however, is exceedingly small in one particular case, being as low as 0.011 per cent. The evil action of even this small quantity and of small accompanying traces of sulphide of ammonium is readily perceived in the sugar liquors which have been run through new charcoal, which should never be used until after it has been well washed and reburned. In addition to the above injurious substances, new charcoal always contains a small quantity of calcium sulphide, and gives off sulphuretted hydrogen when acted on by an acid, and even when only moistened with water. In one instance Wallace found 0.08 per cent. of hydric sulphide in a sample of new char after treatment with acid. The small particles of metallic iron always introduced into animal charcoal from the bones (unless separated by a magnetic separator before grinding, such as that made by the Hardy Patent Pick Co., Sheffield), and also from the grinding machinery, may be separated and estimated by the magnet. It is sometimes blamed for darkening the sugar where such darkening is clearly due to the action of acid sugar liquors on the structural iron of the charcoal filters.

*Presence of interstitial or occluded gases in new and old char.*—Both new and old chars retain within their pores appreciable quantities of gases, which escape when tanks containing them are filled with liquor. Moreover, these gases have been known to explode at the approach of a light to the top of the tank.

2. *Substitutes for animal charcoal.*—Attempts have been made from time to time to mix clay with some one or other form of carbon, so as to produce a mixture which would compete with animal charcoal. The charcoal from certain sea-weeds is said to rival that from bone, but it does not fulfil all the requirements indispensable to a sugar decoloriser.

3. *Basic acetate of lead and sulphurous acid (Scoffern's process).*—Other purifying and decolorising agents, besides lime, blood and animal charcoal, have been suggested from time to time; amongst these, mention must be made of Scoffern's basic acetate of lead process. The prime object of sugar refining is the removal of impurities and colouring principles. Not many, if any, reagents are more efficient in doing so than the basic acetate of lead. Although this fact was well known to chemists, the process was not available owing to the difficulty experienced in removing the excess of the poisonous basic acetate of lead without simultaneously injuring the sugar. Dr. Scoffern, however, accomplished this in a very ingenious and effective manner by forcing sulphurous acid gas through the lead-treated sugar solution by means of a force-pump. By this means every portion of the lead was said to have been removed. In regard to this process the late Professor Brande said: "I do not appear here as the advocate of Dr. Scoffern's process or any other, but it is only just to say that the opinion I gave two years ago in favour of the efficiency of this process and of its absolute safety, when carefully and skilfully conducted, has been fully borne out by my experience since that time. I believe the means of extracting the last portions of lead by Dr. Scoffern's process are infallible, and that they involve no greater amount of difficulty or skill than may be commanded by every sugar manufacturer who chooses to devote adequate attention to the subject." However authoritative Dr. Brande's opinion may have been, and however entitled it may have been to respect, the Government of the day, after exhaustive enquiry by a special committee prohibited the process.

4. *Sulphurous acid.*—Sulphurous acid by itself alone has been repeatedly tried as a sugar-bleaching agent, and, as was long ago pointed out by Dr. Wallace, processes are published every two or three years in which its use is advocated—each writer apparently unaware that his results are well known. At its best, sulphurous acid, according to Wallace, only removes about three-fourths of the colouring matter, and the liquor requires to be treated with charcoal, just as much as if sulphurous acid had not been used. Again, Wal-

lace points out that although it is true that sulphurous acid does not alter cane sugar, like most other acids, yet its liability to change into sulphuric acid involves the risk of introducing a dangerous element; and although the acid itself may be neutralised by lime, the resultant compound formed by the combination of the lime with the sulphuric acid, namely gypsum, is very injurious to the charcoal (by which it is intercepted) in subsequent operations, and renders it less efficient after reburning, if not even injurious owing to the formation of calcium sulphide. But the practice now is first to lime and then to sulphite the limed juice, see pp. 278-280.

The danger of oxidation is not however removed by this revised if new way of working.

5. *Decolorisation by ozone*.—It has not been found practicable to decolorise saccharine liquors by ozone, and the opinion has been expressed that, even if found to be economical in bleaching the colour, it would not be possible to dispense altogether with animal charcoal. Moreover, it has been anticipated that its oxidising action would prove troublesome. Beanes, however, claimed that ozonised air passed through a coloured syrup for three hours exerts a decolorising action as great as that obtained by leaving the sugar in contact with animal charcoal for twenty-four hours.

6. *Defecation of cane sugar by the double carbonatation process as applied to the sugar beet*.—The double carbonatation process so successfully applied on the Continent to the refining of beetroot sugar, has been but little applied on the large scale to the process of refining raw cane sugar. Moreover, it is not anticipated that its application would be attended with any great advantage. As a matter of fact, the sugar solution has to be rather dilute before this process can be applied, and in consequence it would have to be boiled down before being passed through the charcoal, as it is not by any means a substitute for charcoal, but is only supplementary to the action of the latter. Experiments in this direction tended to show that the colour of the cane-sugar solution was little or at all improved. But, on the other hand, the liquors obtained are particularly clear and bright. In certain special cases, however, such as in purifying the washings of animal charcoal, which are highly impure, indeed, and especially troublesome, as well as bag-filter washings and the other impure liquors obtained during the various stages of the refining process, the double carbonatation process might be advantageously adopted in the refining of cane sugar.

Wallace, indeed, recommended that such impure saccharine liquors as bag-filter and char washings and low syrups, much contaminated with salts, should all be purified by precipitating the sugar as a sucrate of lime, and then separating the base by carbonic acid gas; or, in the case of baryta, by sulphurous acid gas. In this connection he suggests as an excellent arrangement to have con-

nected with every large refinery a smaller one immediately adjoining it, where all impure products are treated and worked up apart from the more pure sugars made in the refinery proper. He thus in a way foreshadowed Steffen's process (see *sucrate annexe*, Molasses, p. 227).

*Details of the process of decolorising solutions of raw cane sugar by filtration through animal charcoal.*—The solution of raw sugar, after having been rendered clear, limpid and transparent by being passed through the bag filters (Fig. 101, N), is run into iron tanks or cisterns filled with bone black (Fig. 41, p. 110, and Fig. 101 O). These tanks vary greatly in size and shape; some of them are square and shallow, whilst others are of great depth, attaining in some cases a depth of forty to sixty feet. The form most generally used is that of a circular iron cylinder, say about nine feet in diameter and sixteen feet deep. Such a cylinder will contain about from twenty to twenty-five tons of charcoal, according to its density. This cylinder is packed with the granulated charcoal, which rests on a false bottom of stout linen cloth supported on perforated wood-work. The charcoal generally extends from the false bottom upwards to within a certain distance, some two or three feet, of the top end of the cylinder. The remaining space serves as a reservoir for the sugar solution, which gradually percolates through the charcoal and is allowed to escape into a receiver. In some cases the cylinders are open at the top (Fig. 41), whilst in other cases they are closed as in the Clyde sugar refineries, except the necessary hole for the insertion of inlet and exit tubes and manholes for charging and discharging the animal charcoal, and cleaning the tank and filtering arrangement generally. The tanks are substantially built of wrought iron, and constructed to bear the pressure of a considerable volume of water, which may be applied when necessary so as to expedite filtration.

*Amount of charcoal required to decolorise given weight of sugar.*—The weight of charcoal required for a given amount of sugar is very variable. It depends to a great extent, as has been pointed out by Wallace, on the greater or less abundance of water and on the price of coals, and whether or not the charcoal has to be sent to a distance for reburning. In the latter case, and where coal is dear and water scarce, the quantity of charcoal is reduced as far as practicable, but under more favourable circumstances the ratio should not be less than 25 cwt. of char to a ton of sugar.

*Size or grist of the granulated charcoal.*—This greatly depends on the dimensions and shape of the cisterns, but where practicable, small-sized granules should be selected. Looking at the question from a theoretical point of view, the smaller the grist the better should be the result; but, as a matter of fact, the charcoal granules must be sufficiently large to leave a passage for the liquor to pass through it in something like a reasonable time. In actual practice,



such a size as would pass through a sieve of twenty meshes to the inch, but would be kept back by one of thirty meshes to the inch, has been found to answer best. The use of char dust, which Barbet says is four times more effectual in decolorising sugar liquors than char granules, in beet-sugar factories, has lately been tried *in conjunction with mechanical agitators and filter presses* with good but varying results, depending on quality of juice.

The sugar liquor is run from the blow-up pans into the iron tanks filled with animal charcoal as described, where it is allowed to stand for some hours. It has been found by experience that the *most suitable temperature* for the charcoal to exercise its greatest decolorising effect on sugar is for the liquid to be run off from the blow-up pans at 180° F., whilst the charcoal cisterns should be kept at about 155° F., and never below 150° F. After the sugar solution has been left for a sufficiently long time in contact with the charcoal it is slowly run off from below, while more of the dark-coloured liquor automatically flows on to the top through the ball valve (Fig. 41), so that the tank is continually full. The liquor which comes away at first is not only bright, but colourless as water. Gradually, however, as the process goes on, the liquid becomes darker, until after a certain time it becomes distinctly yellow or sherry colour. The sugar solution is then replaced by the syrup from a previous refine.

*Charcoal washing.*—The syrup is then washed out with hot water. This is an operation of some delicacy. The hot water is run in and allowed to percolate the mass until no appreciable trace of sugar can be found in the washings, that is, until the escaping fluid marks zero, or nearly so, on the saccharimeter—a sure sign that all, or nearly all, the sugar has been washed out of the charcoal. If this operation, therefore, has been skilfully conducted, little or no sugar will remain behind. The great object of the refiner is to remove a maximum of sugar with a minimum of water. The charcoal is now further washed with copious volumes of boiling water, then with cold water, after which it is taken from the tanks and transferred to the kilns to be reburned. The minimum amount of water used in refining 100 tons of sugar is:—

WATER USED IN REFINING 100 TONS OF SUGAR.

	Tons.
Dissolving . . . . .	50
Washing to produce sweet washings, afterwards boiled down or used for dissolving . . . .	40
Extra washing to further purify char . . . .	125
	<hr/> 215, or 50,000 gallons.

*Loss of sugar in bone black.*—Mr. Spencer, in his report on diffusion working *versus* milling working at Magnolia, says: "In

making these grades of sugar, necessitating a double filtration of the syrup, double the loss was experienced in the bone-black room, due to the absorption of sugar by the char and the losses in the waste waters. The filters were open Dumonts (Fig. 41). Owing to the difficulty of properly washing the char without employing a very large quantity of water, the waste products contained considerable sugar, which was daily from 21st November to 24th November, both inclusive, as follows in *sucrose per cent.* respectively, viz., 2.82, 0.91, 1.12, 0.44, 0.73 and 0.91. The normal loss is given on page 305. These analyses showed an enormous loss in the filter-room. The filter-men were instructed to wash their filters considerably longer. Samples were frequently brought to the laboratory for examination, and when the proportion of sucrose exceeded .50 the washing was continued. It is impossible to estimate the loss from this source, but it was certainly very large. It was impracticable to vary the mode of work in the bone-black room. The quantity of waste water was variously estimated at from 1,000 to 1,500 gallons per twenty-four hours; this shows losses ranging from less than 100 to nearly 300 lb. of sugar per day, or from 0.5 to 1.5 lb. per ton of cane. As the amount of waste water could not be ascertained, these figures are a rough approximation of little value."

The *suspended* matter in raw sugar liquors is removed in the process of bag filtration. It is the objectionable *soluble* substances in the raw sugar liquors that are removed and retained by the char. The first thing to be done, then, is to wash out the sugar (as already described) which has penetrated into the pores of the char. But a serious difficulty presents itself. The bulk of the impurities retained by the char are far more soluble in the hot water used for washing than in the original strong raw sugar liquor. They are therefore partially removed in the wash-water. The impurities of the original raw sugar liquor are therefore concentrated in the weak saccharine liquor obtained by exhausting the char, by washing with *boiling* water to such an extent that it is far more impure than the original sugar liquor from which they had been extracted. Vegetable albumen, gum, salts of lime, potash, soda and iron are eliminated from the char by washing, and are concentrated in the liquid along with but a small quantity of sugar. The usual way of working up this *sweet water*, extremely liable to fermentation, is to use it up in dissolving fresh raw sugar, but it is hardly possible to make a greater blunder in sugar refining. This has been aptly described as first taking dirt out and then putting it back in its place, to be again removed with the next accumulation. The free oxygen in the water was at one time said to act under the influence of the bone black upon the vegetable albumen and to change it into a ferment which quickly converts the sugar into lactic acid, and this acid dissolves from the char both lime and iron. The char washings are therefore

very often sour and putrid and heavily charged with lime salts ; and not only so, but they frequently emit a perceptible smell of sulphuretted hydrogen. When the sugar has all been washed out of the char by prolonging the washing, as already described, until the char cedes but little more to the wash-water, it is then drained, taken out of the cisterns, and transferred to the kilns (Fig. 43, p. 117). The usual form of kiln comprises a series of vertical cast-iron pipes about five inches in diameter, arranged in six rows of about ten pipes in each row. Three rows of these pipes are placed on each side of the furnace. They pass vertically through a brick chamber, and are kept at a red heat by furnaces underneath, the products of combustion being drawn off from the sides of the kiln. The pipes are open at the top, where they pierce the brickwork. Underneath they end below the fire chamber in flat sheet-iron prolongations, called cooling boxes, of the same length as the row of pipes to which they are attached, about six or eight feet deep, and an inch or three-quarters of an inch wide, and cooled by simple contact with the surrounding air. In this way the temperature of the char is reduced below the ignition-point before it comes in contact with the superincumbent atmosphere. The wet char, as brought from the filtration tanks, is laid on the top of the kilns, and raked down into the pipes, in which it sinks gradually down as the burnt char falls into the cooling box underneath. From time to time, say every twenty minutes, the bottoms of the coolers are opened, and the char in them partially removed, in such proportion that it will have occupied six to eight hours in its passage through the pipes. The exact time will, of course, depend on the heat of the kiln, and different amounts should be drawn from each individual row of pipes, according to their proximity to the source of heat. If there be three rows of pipes, the row next the fire should be discharged in, say, five hours, the middle row in about seven and a half hours, and the back row in ten hours. Those kilns economise fuel fairly well, but one of their great defects is that the wet mass of superincumbent char forms an obstacle to the free passage of the steam and gases evolved from the simultaneous drying and carbonising of the vegetable impurities in the charcoal. Four-fifths of the heat consumed in the kiln are spent in drying. The char should thus be dried more or less before running it into the pipes. When the charcoal has sufficiently cooled, it is again run into the animal-charcoal tanks. The volumes of water capable of being absorbed and retained by the same quantity of old and new charcoal differ in a very decided manner indeed. For instance, if a funnel be filled with good, perfectly dry new charcoal, and water poured on to it as long as it is absorbed, it will be found to retain in its pores from 80 to 100 per cent., while old charcoal only absorbs from 30 to 45 per cent., according to the quality. Moreover, dry new charcoal does not become

perceptibly wet unless at least 20 per cent. of water is added to it, while old charcoal becomes wet with even 5 per cent. It will thus be seen that the practical man need experience no difficulty in differentiating between old and new charcoal. A quantity of new char was exposed by Wallace to a rather strong heat for an hour in a covered crucible, with the following results: New charcoal 48 cubic feet; after heating one hour, 43·2 cubic feet; after other four hours, 38·0 cubic feet; after still another four hours, 38·5 cubic feet, all per ton. The char thus lost in eleven hours as much of its porosity as it would by being worked in a sugar house for two years. Calcic phosphate fuses at a great heat, but the temperature of a charcoal furnace is only sufficient to produce agglutination.

*Differentiation between old and new charcoal.*—New charcoal burnt till white has the appearance of little pieces of chalk, but old charcoal has the semivitreous compactness of porcelain or flint.

*Changes in composition on repeated reburning and re-use of char.*—*Increase of carbon.*—*Evil effects.*—Old char differs in composition from new char. The proportion of carbon always increases, and if the kilns be perfectly tight this increase is quite rational. In this way the pores of the char are gradually filled up with a deposit of carbon, due to the carbonisation of the organic matter extracted from the raw sugar liquor and retained by the char. This ever-increasing deposit of carbon is an unmitigated evil in sugar refining. Every effort should be concentrated to abate it by thoroughly washing the char by boiling hot water before reburning.

*Decrease of carbon.*—The proportion of carbon in some refineries would not appear to augment, whilst in others it rapidly diminishes, so much so that in exceptional cases it eventually does not exceed more than 2 or 3 per cent.

*Due to access of air.*—Such a great loss of carbon as this can only be due to faulty arrangements at the kilns. Air must find access to the char in the pipes through flaws, etc., but with ordinary care this should easily be prevented, as it is by no means easy to burn off the carbon from the char that has been used for sugar-refining purposes, so as to produce a white bone ash available for making a white or grey coloured superphosphate after treatment with sulphuric acid. (The farmer shrugs his shoulders, or perhaps expresses his disgust more forcibly, at a manure of the colour of boot-blackening.) To prevent air getting access to the hot char, the pipes and cooler boxes must therefore be kept as air-tight as possible.

*Loss of carbon by overheating.*—Loss of carbon may take place by the pipes getting overheated so as to cause a reaction between the carbon and the elements of water, with formation of carbonic oxide and marsh gas. But if the pipes and cooling boxes are air-tight, and the char be not subjected to undue heat, the carbon must perforce increase rapidly, if all, or nearly all, the organic matter

absorbed from the sugar liquor be not regularly and systematically washed out of the char each time it is used before reburning.

*Removal of mineral salts from the charcoal.*—One of the great benefits derived from copious washing of char is the removal of mineral salts absorbed by it from the raw sugar liquor. All raw sugars contain a certain proportion of mineral salts, varying from  $\frac{1}{2}$  to about 1 per cent. in the general run of cane sugars, from 1 to about 2 per cent. in syrup sugars, and in beet sugars from  $1\frac{1}{2}$  to 7 per cent. Highly soluble salts, such as those of potassium, have little or no effect upon char. But they are a source of great annoyance to the sugar refiner, by accumulating in the syrups, from which they can, however, be readily eliminated by a very elegant process patented by Newlands. The more imperfectly or but slightly soluble salts exert a very injurious effect upon the char, unless removed by copious and thorough washing. The most objectionable in this respect is calcium sulphate, a salt only but very little soluble in water. This salt is readily abstracted from the sugar liquor by the char, and, moreover, it can only be eliminated therefrom by copious washing. A very peculiar point about the absorption of calcium sulphate by charcoal is that it is only absorbed by the char from the strong sugar liquor, to be ceded to the wash-water, in which it is carried away to such an extent that an abundant crop, not of sugar crystals but of gypsum, is often obtained in boiling down weak char washings (see Table XVIII., p. 70).

*Bad effect of permanently hard water when used to wash charcoal.*—With a permanently hard water containing much sulphate of lime in solution—saturated with it, as it were—it is almost altogether impracticable, to effectively wash the charcoal, and instances are not unknown of chars contaminated in this way with sulphate of lime to the extent of  $2\frac{1}{2}$  per cent.

*The use of lime involves treating the char with acid.*—Again, where lime is used in clarifying the juice, especially beet-juice, for which it was formerly employed in such quantities, the pores of the char quickly become clogged or choked up with carbonate of lime so as to destroy its beneficial effect on the raw sugar liquor. The only way to restore some of its pristine properties to the char is to treat it with an acid to remove the excess of carbonate of lime (p. 114).

*Influence of repeated use and reburning on the density, volume and porosity of animal charcoal.*—As char ages from repeated use and reburning, its volume gradually contracts or shrinks to a very appreciable extent, so as to diminish or even completely obliterate the pores. According to Wallace, the space occupied by a ton of new charcoal when dry is usually about fifty cubic feet, but after being a few months in use it is reduced to forty, and so it goes on shrinking until it reaches twenty-eight cubic feet, which out of 400 samples examined by Wallace was the most compact. But the specific

gravity of the char is in no way increased. The density, in fact, of old and new charcoal differs but little. New char gives 50·6 cubic feet per ton, old char 35 cubic feet. New char has a density of 2·822, old char of 2·857.

The heat to which the char is subjected is said to produce a semifusion of the calcic phosphate the most abundant constituent of char, and causes a shrinking in the bulk of the particles. This, however, can hardly be, because any actual shrinking of the substance of the char, as distinguished from the coalescence of the particles to form, as it were, one united whole, must increase the density.

*Amount of char per ton of sugar.*—The exact amount depends greatly upon the quality of the sugar to be refined, and also upon the quality of the charcoal. It stands to reason that the smaller the amount of char used consistent with effectual working, the better. The larger the amount of char used, the greater is the loss of sugar, but it also involves the production of abnormally large quantities of weak, highly impure, wash-water. As already mentioned, twenty-five cwt. of charcoal to one ton of raw sugar are quite sufficient if the sugar be of good quality. With fine sugars, equal quantities of sugar and charcoal suffice. One of the greatest mistakes that can be made in sugar refining is to imagine that a *large* quantity of bad or spent char will do the same work as the *ordinary* amount of good char. In the first place, not only does it occupy a great deal more space and thus curtail the yield of refined sugar, but, however large may be the quantity of char used, it does not accomplish the end in view nearly so well. Moreover, it yields an overwhelming bulk of sweet water, *i.e.*, char washings. Working on the large scale, it has been found in actual practice to be impossible to eliminate all the sugar from the charcoal by washing, so that it would be worth while to recover the sugar by evaporation, concentration and crystallisation. Again, there is a loss of 0·75 per cent. of sugar on the charcoal used, *i.e.*, for every 100 lb. of char employed  $\frac{3}{4}$  lb. of sugar is lost. If an amount of char equal to that of the sugar be used, the loss will be 0·75 per cent., whilst if the amount of charcoal be double that of the sugar, the loss will be 1½ per cent. of sugar from this cause alone.

The following analyses of samples of old or used charcoals are due to Wallace :—

TABLE LVII.—ANALYSES OF VARIOUS OLD OR USED BONE CHARS.<sup>1</sup>

	D.	E.	F.	G.	H.	I.	K.	L.	M.
Carbon (nitrogenous) .	9.74	10.60	12.86	19.64	7.42	10.64	5.82	17.28	2.56
Calcic phosphate .	82.80	88.20	81.80	78.20	87.08	80.66	77.26	79.56	90.73
Calcic carbonate .	5.92	4.15	2.92	3.18	1.92	4.52	14.66	1.05	3.50
Calcic sulphate .	.67	.64	.42	1.12	.95	2.24	1.03	.59	1.10
Ferric oxide .	.33	.55	.67	.68	.85	.72	.21	.69	1.17
Silicious matters .	.54	.86	1.33	2.20	1.78	1.32	1.02	.83	.94
	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00
Cubic feet per ton .	44	39	36	32	29	35	40	34	35

D is first-class charcoal. E is excellent. F, fair average quality. G, pretty old and very much glazed. H, very old and over-burned. I has been used in a sugar house where hard water is employed. K has been used in continental beet factory. L has been soured in washing; and M has been exposed to the air while cooling.

*Remedying the effects of continued use and reburning on charcoal.*—So that the char may do its work effectively, repeated renewal of the charcoal is thus very obviously necessary. It will not do to simply replace the char dust sifted out and eliminated from time to time. No more will it suffice to make good the deficiency due to continual shrinkage on reburning by adding new char. The char will not do its work effectively unless an aliquot part of the whole char, and not simply the dust alone, be removed and its place filled up with quite new char. This should be done at the rate of 50 per cent. per annum, and the removal and addition made continuously—one, two or three bags of new char to every cistern each time it is charged with reburnt char.

*Effects of the quality of the water on the composition and decolorising properties of the charcoal after continued re-use and reburning.*—The small percentage of carbonate of lime present in new char is useful in neutralising the small amount of acid present in nearly all raw sugars. Moreover, there is always a certain amount of acid formed during the washing of the char by a kind of fermentation process which it is extremely difficult to prevent occurring. It is not always desirable to use char from which the whole of the carbonate of lime has been removed. The use of such a char will certainly result in the generation of sour liquors, and give rise to iron in the syrups (see pp. 301 and 307). When a very soft water is employed to dissolve

<sup>1</sup> Analyses of new chars are given on p. 295.

the sugar, and also to wash the char, the percentage of carbonate of lime gradually diminishes, and as the char ages it may descend as low as  $1\frac{1}{2}$  per cent., or disappear altogether. With very hard water the carbonate of lime either diminishes to an almost imperceptible extent, or increases often to an alarming degree. In the Continental beetroot-sugar factories, where lime is abundantly used in defecating the juice, serious inconvenience results. Many devices and schemes have been tried to free the char from this obnoxious ingredient. When it is allowed to accumulate in the charcoal to too great an extent, it closes up the pores of the charcoal, so that it cannot fulfil its functions.

*Washing with dilute hydrochloric acid.*—One remedy is to wash the char with 1 or 2 per cent. of hydrochloric acid previously diluted with enough water to saturate the char (see p. 114).

*Beanes' process.*—*Treatment of the char with dry hydrochloric acid gas.*—Beanes' process is based upon the fact that, whilst dry hydrochloric acid passed over dry carbonate of lime remains passive, *i.e.*, it does not give rise to any chemical action whatever, yet when the same dry acid gas is passed over char containing carbonate of lime the latter is completely decomposed—the more so if the charcoal be warm. The freshly burnt char is impregnated with perfectly dry hydrochloric acid gas until the char is thoroughly saturated. The char is then exposed to the air, until the excess of gas escapes, thoroughly washed with water and again burnt. Good results follow the treatment of the char with acid in beet-sugar factories, and also in some particular instances in cane-sugar refineries, where the liquors are for some reason or another slightly alkaline. But in those refineries where soft water is employed the treatment of the charcoal with acid is much to be deprecated, because carbonate of lime, far from being present in excessive quantity, is not present even in sufficient amount to neutralise the very small quantity of acid present in the raw sugar. Although acid-treated char yields a paler filtrate than char which has not been acted on by an acid, yet it is not practicable to remove every trace of acid from the char by simple washing with hot water. There is thus great liability of the sugar in subsequent operations—during the boiling-down process—being converted into fruit sugar. The char washings will be very sour, and the syrups liable to contamination with iron salts (pp. 301 and 306). Working in the ordinary manner, therefore, a smaller amount of syrup will be produced than if the char had been treated with an acid. The sugar from the acid-treated char would, however, possess a better colour.

*Advantage of applying Beanes' process to new char.*—Beanes', and processes of a like nature, may be advantageously used to remove injurious bodies in new char, so as to bring it immediately into a fit working state. Traces of ammonia, and sulphide of ammonia,



as well as free lime and excess of carbonate of lime, are always present in new char. Although the washing and reburning, to which all new chars should be subjected, remove ammonia, carbonate of lime and free lime, yet the excess of carbonate of lime imparts a very yellow colour to the filtered liquors, and generally some five or six weeks elapse before the char is in prime condition. But by adding the new char gradually in small quantities at a time there is little or no risk of any bad result; in fact an immediate decided advantage accrues. For effect of bad washing of char see p. 272.

*Sugar boiling in vacuo.*—The clear limpid syrup from the bone-char filters is then boiled in vacuum pans similar to those used in beet-sugar works (Figs. 54, 55, 100 and 102), but made entirely of copper. The boiling into refined sugar should yield fine regular crystals only. Boiling is done hot, that is, with a slight vacuum, and rapidly.

*The theory of ebullition.*—A liquid boils when the disruptive force of heat on the particles of the liquid is sufficient not only to dis sever them, but also to enable them to overcome the atmospheric pressure, and to cause them to be evolved in the form of vapour.

*Boiling-point.*—The greater the atmospheric pressure, the higher is the temperature at which the liquid boils. The lower the atmospheric pressure, the lower is the temperature at which the liquid boils. The temperature at which a liquid boils is called its "boiling-point". But the "boiling-point" of any given liquid loses much, if not all, of its significance if the pressure of the superincumbent air in which the experiment was conducted be not stated. The atmospheric pressure therefore retards ebullition to a greater or less extent, varying with the altitude. The air is more rarified at the summit of Mont Blanc than it is at the bottom of the valleys underneath—so much so, in fact, that, as every one knows, water boils on the top of that mountain at so low a temperature that it is impossible to cook a potato at that height. But we are not so much concerned with the results obtained by boiling substances in water, but rather with the temperature itself at which the water boils. The pressure of the atmosphere can be removed artificially without ascending a high mountain to a rarified atmosphere. Very volatile liquids, such as ethylic ether, gasolene, etc., can be made to boil at the ordinary temperature of 60° F. by the mere removal of atmospheric pressure.

*Laboratory methods of removing atmospheric pressure.*—This fact can easily be demonstrated by placing a test-tube containing a little ether under the receiver of an air-pump and exhausting the air, when the ether evolves vapour so rapidly as to enter into violent ebullition. This method of facilitating and greatly increasing the development of vapour is in constant use in chemical laboratories. Many substances are greatly injured or even destroyed by expelling

the water in which they are dissolved, by applying heat in the ordinary way. But however delicate a substance may be, the water, or other volatile solvent in which it is dissolved may be easily evaporated from it under the action of the air-pump. Suppose, for instance, it were required to evaporate away all the water of an aqueous sugar solution, and to obtain all the sugar in a crystalline state, the experimental chemist would put the solution to be evaporated under the receiver of an air-pump, along with a shallow vessel containing oil of vitriol. He would now exhaust the receiver of the air-pump, thus causing an increased evolution of vapour from the sugar solution. The vapour might be removed continuously by a prolonged working of the air-pump, but the operation would be troublesome, and the sulphuric acid placed under the receiver renders the continuous working unnecessary, as it attracts and absorbs the aqueous vapour as soon as formed, thus rendering the process continuous.

*The laboratory air-pump inapplicable on the large scale.*—The laboratory method is, for obvious reasons, impracticable on the large scale. If applied at all, it would have to be greatly altered. This was accomplished, in the first instance, in a very ingenious and satisfactory manner by means of the vacuum pan (Fig. 97) invented by Howard, and which is lasting evidence of his constructive skill.

*Description of Howard's vacuum pan.*—The vacuum pan consisted in the beginning of two shells of copper of spherical or spheroidal curve, *s* and *s'*; the edges of both were flanged, and the flanges screwed together.

*The steam jacket.*—The lower of these copper shells was surrounded externally by a steam jacket *f*, by means of which the contents of the vacuum pan may be heated.

*The steam coil.*—A spiral copper worm, ramifying internally, serves as a supplementary source of heat. Through the middle part of the lower copper shell, and through the steam jacket which immediately surrounds it, is an aperture *P*, capable of being shut or opened at pleasure, externally, by means of a valve with ground edges. This aperture is for the purpose of allowing the contents of the vacuum pan, when sufficiently boiled, to escape. Directing our attention to the apertures in the upper copper shell, we find them more numerous.

*Manhole.*—First there is a *manhole m*, exactly similar to the manhole in the boiler of a steam engine, through which a workman enters for the purpose of cleaning the interior, effecting repairs, etc. This large aperture, like the corresponding one immediately opposite in the lower section, is capable of being accurately closed by means of a valve with ground edges.

*Barometers, thermometers and feeding apertures.*—The next apertures to be described in the upper section of a vacuum pan are those

for admitting a *barometer* and a *thermometer* respectively, *b* and *t*, each being inserted through its own air-tight packing. Then comes an aperture through which the sugar solution is introduced.

*How the contents of the pan are sampled without introduction of air.*—*The proof-stick and its functions.*—Lastly comes an arrangement for

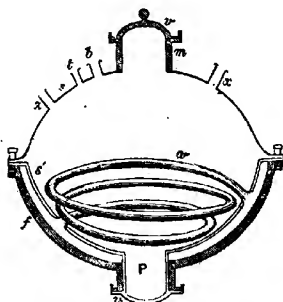


FIG. 97.—Diagrammatic representation of Howard's vacuum pan.

the very ingenious purpose of allowing a portion of the contents of the pan to be withdrawn without letting in air, and consequently destroying the vacuum. It is exceedingly difficult, without actual examination, to give a clear notion of the method by which this problem is solved. A general description, however, is as follows. Into an aperture leading into the interior of the pan one end of a brass pipe is securely fastened (air-tight, of course), and projecting

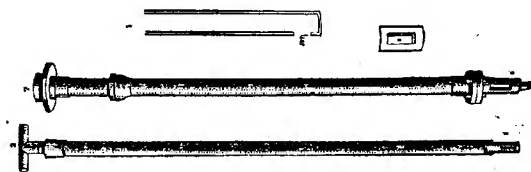


FIG. 98.—Proof-stick. 1, sheath; 2, 2, proof-stick and its spindle; *i*, lateral aperture of proof-stick; *m*, lateral aperture of sheath.

its other end to within six inches of the lowest part of the vacuum pan. This lower end, which when the pan is charged is necessarily covered by the charge, is furnished with a rotary spill like the spill of a cask, and which may be turned off or on at pleasure by means of a key termed the "proof-stick," which projects to the bottom of

the sheath. When turned on, the little excavation *i* (Fig. 98, 2) is made to coincide with the lateral aperture *m* (Fig. 98, 1) of the sheath, and the pan is said to be open. We will suppose, however, the operation of taking a proof to commence when the pan is *shut*—in other words, when the excavation in the spindle does not coincide with the lateral orifice in the sheath. Whilst the arrangement remains as described, the long key or proof-stick is thrust down, its lateral aperture (*i*, Fig. 98, 2) being made to correspond with the lateral aperture of the spindle represented in Fig. 98, 2. The proof-stick being now turned through an arc of two quadrants, both its own lateral aperture and that of the spindle revolving with it are brought into coincidence with the lateral aperture (*m*) of the sheath; and the sugar solution flows into the cup-like depression, or lateral excavation of the proof-stick. Supposing the latter to be now removed *without turning*, the vacuum would be destroyed; but if it be turned back into its original position, through an arc of two quadrants, so as to be no longer in coincidence with the lateral aperture of the sheath, and then removed, a portion of sugar solution will be brought away without in any way affecting the vacuum.

*The wet air-pump; its function and mode of action.*—The liquor is made to flow into the pan by taking advantage of the suction power of the instrument attached to the vacuum pan for the purpose of creating exhaustion, and termed an air-pump (see Fig. 99 and p. 162), although it is not an air-pump in the sense of the instrument commonly bearing that name. It pumps more water than air, and without water it will not act. It may be readily imagined that if a very large air-pump, such as we use in laboratories, only much exceeding the latter in size, were attached to a vacuum pan already containing liquid sugar solution, and that liquid heated by means of the steam jacket externally applied, and of the worm coil internally, the tendency towards a vacuum would be induced, and ebullition at a reduced temperature would ensue. In practice the use of such an air-pump as this would be altogether out of the question. Its size as compared with the ordinary vacuum pan would be enormous. The delicacy of its valves would be such as to render them soon worthless, hence some modifications of the common air-pump is desirable. If we cannot employ an air, or rather a steam, pump, the first suggestion that occurs is, whether the steam might not be condensed into water, and the water pumped away instead of the steam itself? This suggestion has been fully carried out in what is called the *air-pump* of a vacuum pan, and which is in every respect similar to the air-pump of a low-pressure steam engine. It is an instrument of the simplest possible construction, merely consisting of an iron cylinder, into which fits a piston, supplied with one or more metal door-like valves, opening upwards. The action of such a pump as this will be evident from a mere examination of its parts.

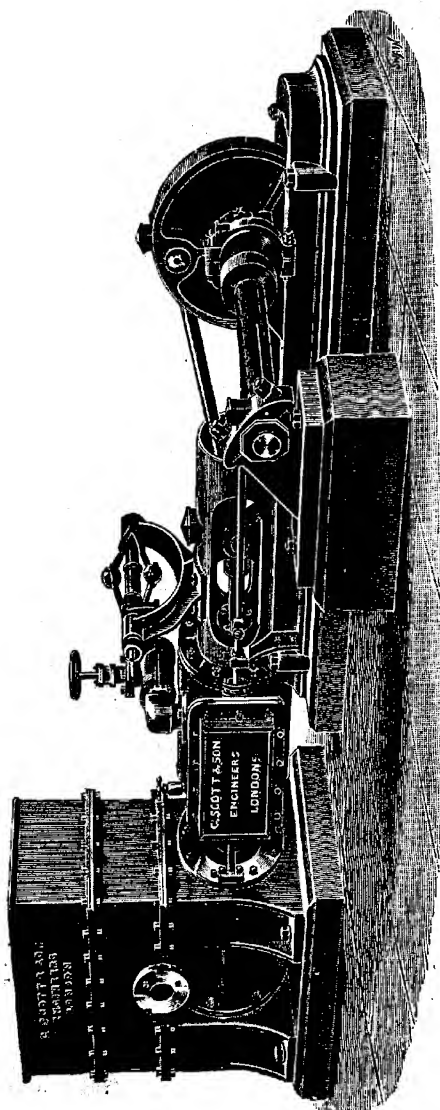


FIG. 99.—Vacuum pump (G. Scott & Son).

The piston being pressed down, the door-valve *will have a tendency to rise*, and, being raised up, the door-valve *will have a tendency to fall*. Now, whether the tendency be brought into active operation or not will depend altogether on the medium with which the pump cylinder may be filled. Any one conversant with the nature of pumps will at once see that such an instrument as this could not pump air effectually, inasmuch as large volumes would pass between the valve and its vent. No less obvious is it that such a pump would be very efficient, provided the object were to pump water.

*Condensers.*—The vapour evolved from the solution under ebullition must pass into an intermediate vessel and become condensed before the so-called air-pump could act. The latter, instead of being directly connected to the vacuum pan itself, is joined to an instrument called the condenser (Fig. 49, p. 162, and Fig. 50, p. 164).

*Injection condenser.*—Condensers assume various modifications both in low-pressure steam engines and vacuum pans. They resolve themselves, however, into two classes, each having its own distinctive type. Either condensation is effected by the injection of water amidst the steam, or the cold water is made to circulate through a series of tubes with which the steam comes in contact externally. Condensers of the former type are the most simple and general. An injection condenser is a very simple instrument, merely consisting of a large cylinder, having within it a tube or tubes communicating with a tank of water externally, and perforated. The consequence of this arrangement is that, as soon as the air-pump is set in action, jets of water escape from these apertures and, rushing amidst the steam, convert it into water now easily recoverable by the pump. From the combined agency of all these appliances, the refiner brings his dense sugar solution to a violent ebullition at a temperature rarely exceeding 150° F.—indeed much lower if required—and thus avoids much of the injurious consequences resulting from the application of heat. But a considerable amount of destruction is effected, even by the vacuum pan, as may be recognised in the final result; for though the liquor originally exposed to evaporation may be, and frequently is, absolutely without colour, the result, after boiling, is more or less yellow, and the amount of tint is always proportionate.

*Practical instructions for using the Vacuum Pan.*

*History.*—According to Dr. Scoffern, ever since the discovery of the vacuum pan in the early part of the nineteenth century, the method of using it was veiled in secrecy. He had had communication at different periods up to 1853 with the greater number of metropolitan, several country refiners, and with a still greater number abroad. Of British refiners, he tells us he had only met

with one principal who had a sufficient knowledge of the vacuum pan to conduct an operation of boiling. By far the greater number were so unacquainted with the machine that they could not check the work of the boiler by reference to theoretical knowledge of the philosophic principles on which the action of the vacuum pan depends. These remarks did not apply to Continental refiners, most of whom even then possessed not merely a theoretical but a practical acquaintance with the vacuum pan, and could use it in the absence of a boiler. It is strange, comments Dr. Scoffern, that the community of "boilers"—most of them Germans—who were engaged in English refineries managed so long to turn their secret to profitable account. They succeeded in creating the impression amongst their subordinates that any attempt to work the vacuum pan without previously making an honorarium of at least ten guineas to the boiler would be futile. Ten guineas was the *usual sum* paid by a pan-man, a sort of sugar boiler's assistant, before he was allowed to take a proof and shown how to put in the grain, this being the technical expression among sugar boilers for the generation of crystals. Nevertheless, it happens that the pan-man may possibly have conducted thousands of boilings, each through nine-tenths of its total duration, for the boiler usually devotes his attention to no more than two phases of the evaporative operation; he stands by, taking proofs until the first crystals have appeared (putting in the grain), and, being called by his subordinate when the pan has got its full charge, he determines when the proper degree of evaporation has been arrived at, consequently when the steam should be cut off and the evaporative liquor allowed to escape from the vacuum pan by opening the valve. Occasionally—but far more rarely than prudence should dictate—a stranger, perhaps a colonist, presented himself at a refinery for the purpose of learning the secret. Then the prescriptive fee of ten guineas was departed from, and the student was charged a sum ridiculously disproportioned to the amount of information conveyed. The vacuum pan is an instrument which depends on a combination of chemical and mechanical principles. Its particular use, too, cannot be learned by mere directions; nevertheless, directions can be so given that any person of ordinary capacity by following them may become, after a few operations, a good practical boiler. The student should consider the nature and objects of the vacuum pan (Fig. 97). It is a copper shell composed of two hemispheroids—the lower one embedded in a steam jacket, the upper one not so embedded. In addition to the steam jacket *f*, external to the lower hemispheroid, there is, internal to the same, and coiling spirally within, a copper steam tube *a*, technically called a worm. In the lower hemispheroid of the vacuum pan there is only one opening *P*. This is for the purpose of letting off the liquor when sufficiently evaporated. It is closed by means of a

valve or door, *opening downwards*, and acted upon by a lever (Fig. 102, D, C). Its edges are ground perfectly air-tight against a rim, and the door should be capable of closing accurately by means of oiled contact alone. Nevertheless, in practice, and with pans that have been some time in use, this accurate contact is not to be depended upon; hence it is a safe plan, and one usually followed, to stop all chance of air leakage by pouring a little syrup into a circular depression, which surrounds the ground edge of the door valve, and supplied with this intention. A similar remark is applicable to the manhole cover *v* and its corresponding rim, to be described hereafter in connection with the upper hemispheroid. These large ground surfaces, in consequence of their great extent, scarcely admit of any other treatment in practice, but it should not by any means be extended to the small ground joints (Fig. 97, *b*, *t*) corresponding with the barometer and thermometer. There can be no excuse for employing, for the purpose of rendering them air-tight, any other liquid than oil sparingly applied.

*The testing of a vacuum pan.—Applying steam.*—It is assumed the operator is about to try the efficiency of a new vacuum pan never yet used. We shall therefore trace the operation of boiling in it a charge of sugar from beginning to end. The first points to be ascertained are the condition of the air-pump and the state of the pan as regards tightness. The operator commences first by setting in action the air-pump; he then turns on a small amount of water into the condenser for the purpose of moistening the valves of the pump, without which care the result of pumping will be inefficient, the so-called air-pump not being intended to pump air, but a mixture of air and water. These directions having been attended to, the barometer gauge is to be carefully regarded. If the vacuum could be rendered perfect (which it cannot), the mercury would stand at the same level in both legs of the barometer gauge. In proportion as such an equality of level is reached or departed from, so will the instrument be in good or bad order. If the mercury refuse to stand, but rise and fall fitfully with every beat and counter-beat of the piston, then there is a leak somewhere. When such a leak depends on a natural imperfection of the vacuum pan, and not on a casualty such as the interposition of a grain of sand or other foreign body in either of the ground joints, then its discovery belongs rather to the engineer than to the sugar manufacturer; nevertheless, for the guidance of colonists and others who may chance to have a vacuum pan and no engineer, a few remarks may properly be given. To determine the exact ground surface to which the leakage is due, it will generally suffice to apply the ear to each in succession, when generally the hissing sound occasioned by the rushing in of atmospheric air will be sufficiently indicative of the leakage. Occasionally other plans must be had recourse to, the



most generally eligible amongst which consists in pouring oil or water externally on the ground joint, and noticing whether it remains there or whether it be forced inward by reason of atmospheric pressure. Should both these means fail to indicate the locality of the mischief, then each joint must be examined in the following manner; indeed, the operation now to be described has to be performed even though the leak has been already referred to one specific ground surface. In the latter case, for the purpose of showing the exact point of imperfection on that surface, we will suppose the ground bearings of the manhole to be examined; for which purpose the ground surfaces are to be well oiled, then the oil wiped off, and the surfaces finally dried by means of a piece of rag or a little cotton waste. A thin paste is now to be made of vermilion and oil, or red-lead and oil, a portion of either of which is to be smeared either on the ground surface of the cover or on the ground surface of the bearing, but not on both. The cover is now to be placed *in situ*, and revolved under pressure in such a manner that every portion of its ground surface may be brought into contact successively with every part of the bearing. The rationale of the treatment with red pigment will now be obvious. If any inequality of bearing exist, such will be evidenced by interrupted spaces, over portions of which the red pigment has become attached, whilst other portions remain uncovered. It is evident that, if the ground surface had been perfectly accurate, the imprint of red pigment would have been equally impressed. It is evident, moreover, that the smeared portions of the ground surface will be those requiring to be lowered in order that the joint shall be rendered accurate. This can be, and indeed is, accomplished by careful filing and scraping. A far shorter process, however, suffices for those small tap-like joints which belong to the barometer gauge and the thermometer (Fig. 97, *b, t*); should either of these joints be imperfect, they may be rendered accurate by means of grinding, a little fine emery and oil being used for that purpose. The operation is conducted by smearing the tap-like portion of the ground joint with the oil and emery, then rotating the latter in its sheath until the joint has been rendered perfect, which may be ascertained by means of the red pigment. We shall now suppose the pan to be proved to be in good order, so far as relates to external appearance; the next point to be investigated is whether it be tight as regards steam. The act of turning on steam is one which demands a few remarks relative to precautions necessary to be taken in order to guard against rupture or explosion. In the first place, it may be laid down as a general rule that a steam tap, with whatever vessel communicating, should never be turned suddenly and at once, but gradually. Neglect of this precaution often leads to dangerous accidents, and always exposes the pipes, joints, and vessels immediately concerned to risk of fracture. There

is another precaution to be observed in the operation of letting steam into the jacket or worm of a vacuum pan, or indeed into any closed vessel. Simultaneously with the entrance of the steam, the air contained in such vessel must be allowed to escape through a tap supplied with this object. The rationale of this operation will be evident on slight consideration. It is this: the steam (always under pressure) enters the closed air vessel, and, disposing of a portion of its latent heat without itself getting less hot, heats, and consequently expands, the air contained in the vessel. Hence the latter has to retain the pressure, not of steam alone but of a mixture of steam and air. Consequently, before turning steam on to the vacuum pan, or any vessel constructed on a similar principle, it is always necessary previously to open an air-tap, for the purpose of allowing air to escape. The tap should be retained open until steam is observed to issue violently from it: it should then be closed. The practice of retaining it a little open during the whole operation of boiling, as is followed by some sugar boilers, is totally unnecessary.

*Pressure of steam.*—As to the pressure of steam best adapted for working the vacuum pan, in the greater number of sugar houses it is not allowed to exceed 10 lb. to the inch; it may be that a still lower pressure, say 7 or 8 lb. to the inch, is even better. At any rate, steam of above 10 lb. to the inch should not be employed: firstly, because its heating power (under the circumstances of its employment) is not so great as that of steam at a lower pressure; secondly, because of the dangerous strain to which high-pressure steam subjects the apparatus. In stating that high-pressure steam has not so great a heating power as low-pressure steam, the remark must be limited to the condition of heating a fluid by means of a steam-warmed jacket or coil. If a fluid be heated by injecting steam into and through it, then high-pressure steam, weight for weight, is the more powerful. Inasmuch as the greater number of refineries possess high-pressure engines, although low-pressure is necessary for working the vacuum pan, an intermediate receptacle called the expansion vessel is required. It consists of a metallic cylinder, to which a valve is attached, weighted to the degree of maximum pressure which the contained steam is allowed to reach, and which pressure once exceeded, the valve opens and, facilitating the escape of a portion of the steam, maintains the desired equalisation.

*Condense box.*—Proceeding with our investigation of the successive phases of action which develop themselves on admitting steam into the jacket and copper worm of a vacuum pan, we shall presently find the necessity of an adjunct as yet undescribed. The steam portions and appendages of the vacuum pan being only heated to the temperature of the surrounding atmosphere anterior to the admission of steam, it follows that a certain amount of steam will be condensed

into water by the operation of cooling. It is certain, moreover, that such cooling agency will be always in operation; therefore, except some provision were made for removing the water thus resulting from condensation of steam, the jacket and worm would soon be rendered inoperative. Such provision is made by an instrument termed the *condense box*. To describe the mechanism of this apparatus is unnecessary, seeing that, its principle being enunciated, a slight examination of the mechanism of the apparatus will show in what manner the conditions are applied to practice. The "condense box" must necessarily be fixed on a lower level than that of the vacuum pan, from which it is usually some considerable distance removed.

*Testing steam jacket and steam coil for steam-worthiness.*—Let us now return to an examination of the pan itself. We have already determined that those portions of it not having immediate reference to steam are tight and trustworthy; it remains, therefore, to examine the condition of the steam jacket and coil. For this purpose, the steam being laid on with all the precautions already detailed, the operator places a strong light near the lens orifice and looks into the pan through the sight-hole opposite the former; or, turning off the flow of water into the condenser (not to be confounded with the condense box), stopping the air-pump and removing the manhole door, the observer may stand on the rim of the pan and look through the manhole. It is possible to conceive a case of steam leakage under the aid of a vacuum ceasing to be a leakage when the vacuum is removed; such a case, however, is so exceedingly improbable that the examination of a vacuum pan as to steam-worthiness through the manhole may be regarded as perfectly reliable; indeed, on account of the larger and more ready sphere of vision thus commanded, the plan is preferable to an examination of a pan through the sight-hole. Any defect as regards steam-worthiness can easily be observed, and its locality no less easily determined, by noticing the exact point at which the steam escapes. The escape may be either internal or external. It may be from a crack in the substance of the steam jacket or from a defective joint. If the former the defect is fatal; the jacket must be replaced by another. If on a joint, it may be remedied by unscrewing the latter and replacing it with tow and a mixture of red and white lead; if in the worm, the latter must be removed and the defective part made good by brazing. Wherever a steam leak exists, and from whatever cause, it is always to be regarded as a serious impediment; but next to a crack in the jacket, the existence of which renders an explosion imminent, a defective worm is the most grave condition. The consequence of the latter is that a charge of sugar under process of boiling continually undergoes a joint operation of dilution and evaporation, thus *pro tanto* destroying the specific advantages of the vacuum pan.

*The actual process of boiling in vacuo.*—Let us now assume the vacuum pan to have been determined perfectly efficient in every respect, and the process of boiling to be commenced. The operator begins by first setting in motion the air-pump, then turning on the condensing jets, opening the air-tap in the vacuum pan jacket, and gradually turning on the steam. He now begins to charge the pan, keeping correct account, measure by measure, of all the liquor which goes in. Of measures there are two kinds—one kind open, the other closed. The latter is most frequently employed, possessing the advantage of drawing its supply of liquor from a lower floor than that on which the pan is placed, such being in most refineries the place of greatest eligibility for the liquor cistern. It need scarcely be indicated that closed measures, as they are called, communicate by means of a tube with the liquor cistern, through which the liquor ascends into the measure by the force of the suction exercised by the pan itself. The total number of measures of liquor which a vacuum pan is capable of working being known, about one-third of that total quantity should be allowed to enter, and should be submitted to the full force of the pan—that is to say, the act of ebullition may be carried to any extent short of that which would result in boiling over. At this stage no harm can accrue from carrying on evaporation with rapidity, and besides other advantages a considerable saving in time will be gained. It is not to *weak non-concentrated* liquors that even the greatest evaporative power commanded by the vacuum pan can be injurious, but to the *concentrated result* of evaporation hereafter to be described.

*The condensers : their capacity and rational working.*—If the liquor be of good quality the boiler has very little to be solicitous about at this stage of the operation, further than observing that every function of the pan is being well performed. He need take no account of the liquor within. There is, however, one function of great importance in itself, and the consideration of which boilers too frequently neglect—and that is *condensation*. If this be not adequately performed, much of the efficiency of the vacuum pan is lost. In order that condensation may exercise its maximum effect without overbalancing the exhaustive action of the pump on the one hand, or permitting a portion of steam to remain uncondensed on the other, some care is necessary. The condenser should never feel hot. Therefore, if such elevation of temperature be recognised on applying the hand externally, more water should be turned on. If, notwithstanding the supply of the maximum amount of water, compatible with circumstances presently to be described, the condenser is still hot, the steam power of the pan must be diminished by a special turning of the tap. The circumstances acting as a limit to the quantity of water capable of being brought into action by any given condenser are chiefly : (1) Size of the condenser ; (2) number

and size of jets communicating with it; (3) power of the air-pump. If, from any combination of circumstances, the condenser be overloaded with water, the latter, instead of being removed altogether by the pump, flows back into the vacuum pan, and, mingling with the sugar solution, proves very detrimental to the ultimate result. In proportion as the fluid to be evaporated is less dense or more watery, so will the amount to be removed by evaporation be greater; hence the size and power of the condenser must have immediate reference to the purpose for which the pan is intended. A power of condensation amply sufficient for the necessities of a vacuum pan, when employed in concentrating the dense liquors of a refinery, may prove totally inadequate to the altered necessities of colonial manufacture, where the juice to be evaporated is more watery. Scoffern relates on one occasion he conducted the evaporation of cane juice to crystallised sugar altogether by means of a vacuum pan. The condenser was one of great power, and the command of water unlimited; had it not been for these circumstances, the operation could not have been carried on to a successful termination. Returning now to the liquor under operation in the pan, it will be time to examine, by taking a proof, to what extent the evaporation has been pushed. The original quantity of liquor in the pan is supposed to have been a third of the pan's total charge, its density is supposed to have been 28° B. (sp. gr. 1.245), its quality the best. Under these circumstances, supposing the evaporative operation to have been briskly carried on for about half an hour, crystals should begin to manifest themselves. The only certain way of recognising their presence, judging of their size and their general qualities, consists in taking a proof, full directions for accomplishing which will presently be given.

*Decrepitation in pan as crystals begin to form.*—There are other indications, however, of the formation of crystals which the operator should learn to appreciate. One of these is the peculiar crackling sound, like that of salt thrown on the fire, recognisable as soon as crystals have formed, on applying the ear to the side of the vacuum pan. It appears to depend on an alteration in boiling of the solution of sugar in which crystals have begun to form.

Previous to this period it is viscous, *tenacious*; subsequently to the formation of the crystals the physical character of the liquid alters. It is more thin, less tenacious, and the angular crystalline particles which it contains exercise a physical effect on the operation of boiling, which now takes place with greater rapidity than before, and with a series of sharp but minute explosions. This peculiar phenomenon, which may be called *decrepitation*, is especially noticeable where a solution of pure cane sugar is operated upon. The boiler should lose no opportunity of making himself familiar with so important an indication.

*The flash.*—Another sign of the formation of crystals in the liquid undergoing evaporation consists in what is technically called "the flash". This is a sort of sparkling, recognisable on looking down upon the charge through the sight-hole. The flash is due to the operation of certain reflective and refractive effects of the crystals on the beam of light shining down upon them from the lamp.

*Of taking the proof.*—This is the great secret of vacuum boiling, making known to the operator the exact condition of things inside. It is accomplished by means of the very ingenious contrivance termed the "proof-stick". It will facilitate the comprehension of this part of the subject if we treat separately of the method of getting out a proof, and the examination of this proof when got out. The operation of getting out a proof requires four distinct manipulative acts, all of which it is necessary for the practical sugar boiler to comprehend thoroughly. They may be described as: (1) Insertion, or position 1. (2) Half-turn upwards to the left, or position 2. (3) Half-turn downwards ending at position 1. (4) Withdrawal, position 1 being maintained for about the first six inches, then position 2. Consequently it appears that, the proof-stick having been properly inserted, the remaining portion of the manipulations is a matter of simplicity. Now, as regards this insertion, it must always be remembered to thrust down the proof-stick with the scooped portion of its extremity (Fig. 98, i) looking downwards. Of course there can arise no difficulty in respect of this, but frequently it happens that after the proof-stick has been allowed to remain sometime in the sheath the operator forgets in which direction this scooped portion of the extremity lies, whether upwards or downwards, hence an additional means of discrimination is necessary. This is provided by the maker in the following manner. The handle of every proof-stick is marked with a little round excavation on the same side as the scoop at the extremity, whence it follows that, whether the proof-stick be within or without the sheath, the exact position of its scooped extremity can be readily determined.

*The examination of the proof.*—Returning now to the operation of examining the proof withdrawn. Next to the operator is placed a light, a gas light being preferable on account of its brilliancy. Usually this light is placed in a box, the sides of which are painted dark, though this arrangement is not indispensable. The object of the light is to enable the operator to examine the condition of the proof by the transmission through it of luminous rays, by which means, if any crystals are present, they may be readily seen long before they are large enough to be felt. The means adopted for transmitting luminous rays through the evaporated material consist in throwing it out from the scooped end of the proof-stick upon the thumb of the left hand, approaching the forefinger, separating it,

and thus extending the material to be examined in the form of a sheet; this being held between the source of light and the eye, the presence or absence of crystals is readily determined. Supposing the operator to be satisfied that crystals, technically called grains, really exist, he has nothing more to concern himself about just at present, further than to take care that the evaporative operation go steadily on; under which treatment the crystals will soon be found to increase in size and in number; to become tangible as well as visible, feeling between the fingers like particles of sand, which in proportion as they are large and hard, in such proportion are they better—at least, according to English commercial notions. On the Continent, however, the production of small-grained sugar is aimed at, and indeed whiteness, *ceteris paribus*, is in direct proportion to smallness of crystal. At this stage of the operation the practice of boiling differs in the hands of various operators—as indeed it must, according to the quality of material exposed to the evaporative action, and the exact condition of staple required. As a general rule, however, it is proper to carry the evaporative process to such a pitch that a proof can barely be attained on account of the thickness of material in the pan. When this state is arrived at, then it is time to increase the amount of material by turning on a flow of liquor from the measure into the pan.

*Feeding the pan:* (a) *by gushes*, (b) *by gradual infusion*.—There are two distinct methods of feeding the pan—either by gushes or by a process of gradual infusion. The latter is in theory the most scientific; it is that most generally adopted by intelligent sugar boilers, and is in every way the best method. In practice it is no less easy than it is comprehensible in theory, and consists in so arranging the flow of liquid that the amount of liquid allowed to enter the pan may be proportionate to the force of evaporation. If it be less, then the contents of the pan will become too thick; if it be more, then, supposing the operation of evaporation to have been continued sufficiently long, it is evident that the crystals already formed will become dissolved. This latter is one of the most unfavourable state of things which can occur, but it may always be prevented by attention on the part of the boiler, who, by means of frequently taking proofs, can always make himself acquainted with the exact condition of his charge (cp. Fig. 98, p. 310, and context).

*Final precautions*.—When the last portion of liquor is already in the measure, and the operation of boiling almost finished, the boiler must pay great attention to his charge. The quality of his resulting sugar-loaves will in great measure depend upon the boiling operation ceasing at exactly the proper instant of time, a few seconds more or less making great practical variations in the results. Nothing but practice—very extended practice combined with great intelligence—can supply the needful knowledge; by due attention to theory

and general principles, the errors of boiling may be restricted within the range of comparatively narrow limits. Perhaps the following general directions for concluding the operation of boiling are the best, though considerable manipulative discrepancies exist in the practice of different operators. Whether the vacuum pan be fed by sudden gushes or gradual infusion, it is always a safe plan for the boiler to adopt the former, as concerns the last measure of liquor. With whatever degree of care the turn of the liquor-tap may have been adjusted, it will be generally impossible to arrange it precisely at that point conducive to the best final result. At the point when all the liquor save the last measureful has been turned into the pan, and a proof being taken, the operator probably will find that the result is either a little too thin (free), or a little too thick (stiff). Such may happen to charges under the care of even the most experienced boilers. Where the system of gradual infusion is allowed to proceed, this undue stiffness or thinness would continue to the end of the operation; whereas, by taking the last measureful of liquor under his immediate control, the boiler has it in his power to modify this state of things, and bring the evaporative process to the condition finally required. With regard to the exact condition of stiffness at which the operation should be concluded, and the contents of the pan turned off into the heater (Fig. 102, G), there is a considerable difference of practice. Supposing the material to be of the best quality, the operator may be told, as a general rule, to boil up to the degree of thickness at which a proof can barely be taken. Admirable results have been known to be produced from the contents of a vacuum pan boiled so very stiffly that not only could a proof no longer be taken, but the material was obliged to be forced out of the pan by a pole inserted through the manhole. This material, however, was the result of liquor which had been purified by means of lead (p. 297). Sugars defecated by lime and bullock's blood in the old-fashioned manner contain such an amount of glutinous adhesive matter that an evaporation carried to the extent described would have been totally incompatible with the subsequent operations of claying, liquoring and drainage, operations on the successful performance of which the perfection of the final product depends. Immediately the evaporative operation has been carried to the furthest extent desired by the boiler, the process should be stopped. First of all the steam communication is cut off; then the flow of water into the condenser; next, the air-pump; next, an air-plug being removed, or a tap being opened (contrivances vary in different pans), air is admitted into the pan; and finally, the let-off valves C or C' being opened by the lever D or D' (Fig. 102), the charge escapes into the heater G below. At this period a tap is turned, by means of which a jet of steam is injected into the pan, for the purpose of freeing it from adherent sugar.



*Boiling by doubles.*—Occasionally, when the liquor to be evaporated is of inferior quality, the ordinary conditions of boiling already described are not capable of yielding a sufficient crop of well-defined crystals. Under these circumstances the operation of boiling by doubles, as it is technically called, is frequently had recourse to. The theory on which it is based rests on the fact that crystalline nuclei being placed under favourable circumstances in a vehicle containing similar material in solution, crystalline molecules are not only deposited on the nuclei already existing, but the formation of additional crystals is determined. In the operation of vacuum-pan boiling this principle is readily applied by retaining a small portion of one charge as a foundation for the next (see pp. 185 *et seq.*).

*Boiling by the thread proof.*—The operation of vacuum-pan boiling, as already described, has been conducted on the supposition that crystals are formed during the evaporative operation, and the gradual progress of such boiling has been judged of in reference to the appearance of crystals formed as indicated by the proof. Such a result, however, only applies to liquors which are moderately good. In proportion as the material to be boiled becomes laden with glucose and other impurities, so will its tendency to generate crystals during the evaporative act diminish; and, in the course of the actual routine of a refinery, many charges will have to be submitted to the action of the pan which no manipulative skill could succeed in granulating under the operation of boiling; hence the method of judging by proof requires to be varied, seeing that no crystals exist, for the guidance of the boiler. Recourse must then be had to the indication afforded by the length, brittleness and some other characteristics, of a string of the proof, formed by bringing a portion of the evaporated mass between the finger and thumb, and separating them. Three distinct phases of manifestation will be rendered evident under this treatment. The string may be formed of the length of two inches or more. After existing for the period of a second or two the string breaks midway, each end rapidly, almost suddenly, shortens, returns respectively on the finger and thumb and becomes lost in the two portions of the original mass. This indication serves to show that the material has not been boiled enough for the final purpose. Supposing the evaporative operation to be still pursued, the thread, after breaking as before, forms a hook at either extremity, which is indicative of the fact that the evaporative process has been almost carried far enough. The evaporation being still further continued, the string breaks as before, but its extremities form little spirals, each something like the spiral of a corkscrew, the thread still retaining its power, though in a diminished degree, of collapsing on the finger and thumb. Beyond this degree of evaporation the operation should not be

carried; if pushed to such an extent that the collapsibility of the thread is lost, the final result will drain with difficulty or not at all—in other words, the operation will have been a failure.

*Cleansing the proof-stick.*—This point in vacuum-pan boiling is one of such great importance, and the neglect of it will so completely destroy the efficiency of the proof apparatus, that for all practical purposes the latter might as well not have existed. Nothing but the most scrupulous attention to cleanliness will maintain it in good working condition, to say nothing of the chances of accident to the apparatus from attempts to wrench out the proof-stick when it has become firmly set in its sheath by means of consolidated sugar.

The cleansing of the proof apparatus involves two operations: (1) The cleansing of the proof-stick, and (2) the cleansing of its sheath. We shall now give a description of both operations, commencing with the former. The proof-stick having been removed and the proof examined, the operator, by means of a bit of chip, clears out the proof hollow; he then plunges the instrument into a pail of water, withdraws it and wipes it dry. Finally, he oils slightly the two ground or bearing portions of the instrument, namely, the ground ring immediately contiguous to the handle, and the extreme further end. These operations being performed, the proof-stick may be regarded as cleansed, and ready to be employed in taking another proof. The operation of cleansing the proof-sheath is accomplished by sponging—that is to say, a little piece of sponge or cotton waste or soft rag, being firmly attached to the extremity of a rod, is moistened with water, plunged down into the extremity of the sheath, rotated several turns, finally withdrawn, washed, and the operation repeated as often as may be necessary, until the cleansing has been accomplished, when, finally, the external ground socket of the proof-sheath is slightly moistened with oil. In conducting the operation of cleansing the proof-sheath, two special points present themselves for remembrance. The rag, cotton waste, sponge or other cleansing material attached to the extremity of the rod already described must be very firmly attached, otherwise there will be great danger of its remaining in the proof-sheath. Should this ever occur the soft material must be removed at once, which can be best effected by the screw end of a gun ramrod. The second especial point for remembrance is this: the sheath, although it must be perfectly clean, should never be allowed to retain any considerable portion of the water wherewith it may have been washed—that is to say, although it is a good plan to begin operations by thoroughly saturating the sponge of the cleansing stick with water, yet this water, having served its purpose, should be finally removed by insertion, withdrawal and squeezing of the sponge. The propriety of this care will be evident on considering that the operator judges of

the progress of his boiling process mainly by the consistency of his proof. In proportion as the latter is thick or thin, so is it inferred that the evaporative process has advanced or been retarded; but if the water due to the cleansing sponge be not accurately withdrawn

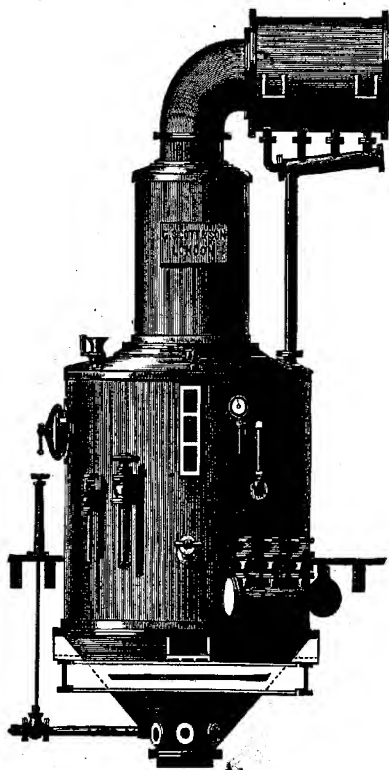


FIG. 100.—Vacuum pan.

from the proof-sheath, it is evident that a fallacious indication of thinness will be manifested.

*Reheaters.*—When the vessel is full, and when it has been found after many trials that the boiling is finished, a valve situated in the

lower hemisphere of the pan is opened and its contents run into a capacious copper vessel (Fig. 102) placed immediately below the vacuum pan, usually on another floor provided with a steam jacket, and furnished with a large tap or a discharge sluice. This vessel is constructed, in fact, like the lower half of the vacuum pan, *i.e.*, it consists of a copper shell enveloped in a steam jacket. It has not, however, like the lower segment of a vacuum pan, a copper coil or worm. The boiling is kept up to about 80° C. (176° F.) the whole of the time it is in the reheaters. Heat is kept up exclusively by the jacket. The vessel which we have just described is termed by some refiners the "heater," by others the "cooler". The reason for this apparent anomaly is to be found in the fact that, whatever be the temperature at which the solution of sugar has been boiled, it requires to be "potted" at a temperature lying between 170° and 185° F. But when the old method of boiling, namely, the open-pan method, was in vogue, it necessitated a final temperature of 230° F., and often even a still higher one. The vessel into which the boiled mass was poured before potting was then in reality a cooler. But since the vacuum pan came into use in sugar refineries for boiling down sugars, the temperature at which the operation is conducted being in most instances lower than 160° F., it therefore follows that the vessel in which this boiled mass is raised to a temperature of 170° or 180° F. acts as a heater. This is the cause of the anomaly in the diametrically opposite terms used to designate this vessel. The rational use of this heater requires much skill and experience. Its scientific use depends on the physical law that there is a certain heat within the comparatively narrow limits of which those solutions which are capable of being crystallised have a maximum tendency to assume the crystalline condition. As it comes from the vacuum pan the boiled mass should have assumed a decidedly crystalline condition during boiling—that is, unless the charge be one of a "low" nature containing large proportions of glucose and colouring matters, and which can only be wrought up into the brown sugars known as "pieces" and "bastards". In any case, the object of the heater is to still further increase the conditions favourable to crystallisation. This is done by raising the temperature up to a certain limit, according to the nature of the charge, good sugar being potted at a lower temperature than poor qualities. In the case of first-class loaves the temperature of 80° (176° F.) is the best. The extra heat required to raise the temperature is got by applying steam to the jacket, and during the time the heat is getting up the mass is kept agitated. The general routine of sugar-house custom is to heat all the boiled masses, whatever their nature and their origin, to the same temperature in the heater, without regard to quality or consistency. This is a bad practice. By modifying the temperature

to which the *masse cuite* is brought in the heater, many defects of

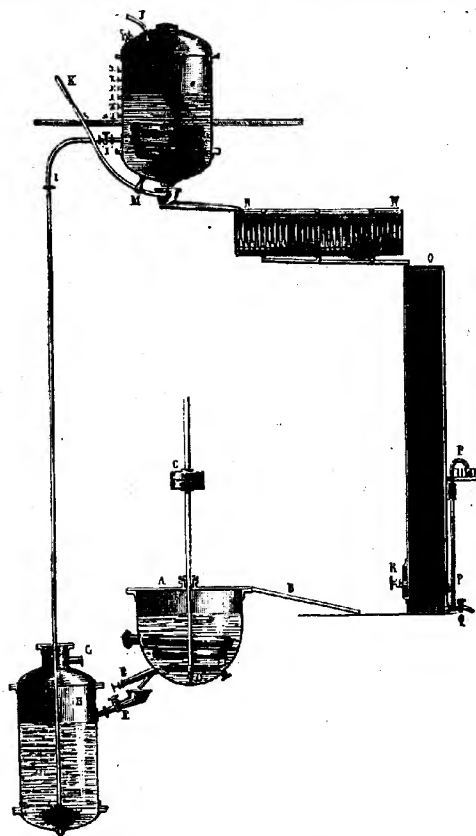


FIG. 101.—Diagrammatic representation of sugar refining.—A, blow-up pan ; H I, montejus ; L, defecation pan ; K M, discharge lever and valve ; N N, Taylor's filters ; O, bone-char filter ; P P, swan-neck for feeding pipes conveying filtered sugar liquor to respective vacuum pans.

boiling, such as too great freeness or stiffness, may be obviated. One of the many conditions favourable to the perfect growth and

development of crystals is perfect freedom and liberty of motion between the particles or molecules about to assume the crystalline condition. But the temperature most favourable to the crystallisation of sugar particles or molecules is considerably higher than that which can be maintained during the whole of the time occupied in boiling without destroying a large proportion of the sugar, the prevention of which destruction is the principal function of the vacuum pan. It is, therefore, customary to boil the sugar solution at a much lower temperature than that most favourable to its perfect crystallisation, and only imparting this temperature for a short time at the end of the process by means of the heater.

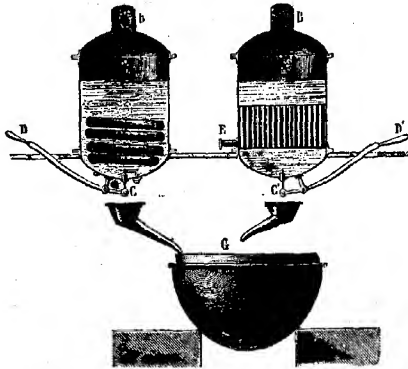


Fig. 102.—A A, vacuum pans; C C', let-off valve; D D', discharge lever; G, reheater.

A diagrammatic representation of the several stages of sugar refining until it reaches the reheater is shown in Figs. 101 and 102.

*The fill-house.*—The moulds for forming loaf sugar are arranged in a capacious building. They are placed on wooden supports, which keep them perfectly in line in the piece.

The moulds (Fig. 103), the profile of which represents a sugar-loaf, such as it is known to every one, are made of iron, painted on the one side with a sort of enamel; at their apex is a hole which is closed by a wooden peg. All these moulds are placed empty in their supports, with the apex pointing downwards. When the boiled mass is run into the reheaters the workmen bring their large beaked basins, shaped something like coal-scuttles, which they carry by straps, and fill them by opening the valve, and then discharge them by pouring them into the moulds, which are thus filled one

after the other with the hot boiled mass. The work of these men is often done by machinery. A trough running on wheels above the moulds fills a whole row at a time, and so on from row to row in succession. The moulds thus filled are soon covered with a solid layer of crystallised cooled sugar. A workman provided with a large wooden knife or spatula, technically known as a hauling knife, breaks the crust and energetically stirs the mass so as to distribute the crystals until the loaf has gained a certain consistency. Sugar-cones over 14 lb. in weight are not considered loaves in commerce, but are known as lumps, titlers, etc. These are not generally subjected to the process of hauling. It is then left to itself. Care is taken to keep the room at a constant temperature of  $30^{\circ}$  to  $35^{\circ}$  C. ( $86^{\circ}$  to  $95^{\circ}$  F.), and especially to avoid air currents or draughts. The moulds and their contents are left to stand at rest for a period of at least twelve hours, except as far as having been stirred in the face during the early portion of this period, so as to make sure that

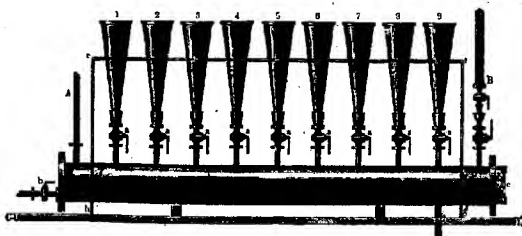


FIG. 103.—Sugar-loaf moulds for draining sugar-loaves by suction.

the crystals form and settle in a regular and uniform manner. During this period of repose the crystals as they form settle, and moreover, as the superincumbent syrup cools, the sugar which it deposits acts as a binding agent to cement the mass into one homogeneous whole. But as the solution of sugar which on evaporation and boiling down yielded the boiled mass was far from being pure, and was moreover more or less coloured, it results, as a matter of course, that all the impurities have been concentrated to a small bulk in the boiled mass, or rather, and in less bulk still, in that portion which still remains in the liquid form. This impure liquid is known as the "green syrup," and must be eliminated from the crystals forming the loaf before the latter can be dried and put on the market for sale. With this end in view, the loaves are transferred by lifts to an upper storey, and, first of all, the wooden plugs which have been used to stop up the apex of the moulds are removed and a pricker or brad-awl is forced up a few inches into the crystalline

mass, so as to make a channel through which the impure syrup may drain itself away. The moulds, unstoppered and pierced or primed in the manner indicated, are now placed apex downwards on racks or tables, troughs pierced with holes of the same shape as the moulds underneath, which are inclined troughs or gutters almost on a level with the floor, which collect the whole of the syrup from many hundreds or thousands of these cones and conduct it into one general reservoir. Here it may once more be usefully observed that, no matter how white or pale the original sugar liquor was before boiling, that operation, even when conducted with the utmost care by means of the vacuum pan at the lowest possible temperature under the most favourable circumstances, brings in its train a certain amount of destructive action and consequent darkening in colour. It will not therefore be a matter for surprise that not only is the syrup which flows from the pierced cone not perfectly colourless, but the sugar itself from which it drains is not completely white even though the coloured syrup which occupied the interstices between the crystals has been drained away. Enough syrup, indeed, remains adhering to the crystals to colour the whole mass a dark yellow or even brown. Each crystal, is, in fact, surrounded by a coating of coloured substances, so obstinately persistent that nothing less than a washing of the crystals will suffice to remove it. This washing process is performed after the loaves have drained for about a week.

Many modifications of the washing process have been tried from time to time. The claying process is the oldest and perhaps not the least effective method. This consisted in the application of *clay magma* to the face of the cones, just as is done in the manufacture of clayed sugar from the original cane juice. This method of washing is known by the name of *claying*, and may well be designated *claying properly so called*, to differentiate the process now universally adopted in sugar refineries, which has also been designated as *claying*, although no clay whatever is used in the process. The "*claying*" process consists in breaking the upper crust of the face or base of the sugar-cone, which is very hard, and in applying to the fresh exposed surface a thick magma or paste of sugar and water. The action of this superimposed layer is similar to that of wet clay. A certain proportion of the concentrated saccharine fluid gradually percolates through the interstices of the loaf, driving the coloured syrup before it. It will be at once seen that, by repeating this process sufficiently often, the sugar-cone would eventually become perfectly white. In actual practice, however, that is not the course pursued: the so-called *claying* process is merely a preliminary to the subsequent process of *liquoring*. The rationale of the liquoring process depends upon the sufficiently apparent principle that sugar at any given temperature is insoluble in water which has already been



saturated with sugar at that temperature. A saturated solution of pure sugar is prepared of a density of 1.291 (83° Baumé; 58° Twaddell), and stored on the top floor of the refinery under the name of "*fine liquor*" or "*magma liquor*". This liquor, not being subject to fermentation, may be stored for an indefinite period. The liquoring process follows the operation of claying. The "clay," or paste of sugar and water, was superimposed on the face or base of the sugar-cone. By this time it will have driven a certain amount of the coloured syrup before it, and the sugar-loaf will gradually have become dry. The workman now smooths and evens the base of the sugar-loaf with his bottoming trowel. He then runs on magma liquor to the depth of some inches, and allows it to percolate through the whole cone. The process of liquoring is an interesting one. The expulsion of the dark-coloured syrup is not the only result attained; the sugar left behind increases in weight by the segregation of fresh crystals coming from the *fine liquor*. Consequently the loaf actually increases in weight instead of decreasing. It is stated that for some reason or other the combined processes of *claying* and *liquoring*, as now practised in sugar refineries, does not succeed except on sugars which have been prepared by the vacuum pan. Hence in those countries where open-pan boiling is still in vogue the process of *claying* with *clay* is still practised.

A single liquoring operation seldom suffices to purify a sugar-cone. Several successive liquorings are given, almost always two, occasionally three, but seldom more than three. The progress of the process is judged from time to time by knocking a loaf out of its mould from a lot under treatment, and inspecting and observing how the percolation is proceeding. Should the crystalline mass present a satisfactory appearance, the cleansing process is delineated by a sharp distinct line. On the other hand, if the boiling process has been unsatisfactorily conducted, or if the sugar was of bad quality to start with, then the cleansing goes on in a very irregular manner. In any case, this washing of the crystals is continued until the loaf is uniformly white throughout its whole mass. A sugar-cone may be ascertained to be free from coloured syrup that is arrived at the stage of the process technically known as *neat* by the appearance of the liquor which percolates from the perforated apex of the mould. A couple of days is now allowed for complete draining. Or, draining may be hastened by transferring the moulds, apex always downwards, to the suckers shown in Fig. 103, pipes with rubber fittings in which a vacuum is made by a pump which sucks out the last trace of now useless cleansing liquor. As percolation begins to cease, the sugar-cones are loosened in their moulds by knocking the moulds against a solid wooden block. When the cones have completely ceased dripping and percolation is finished, the faces of the cones are trimmed or dressed—that is, the loose sugar on the face is cut off,

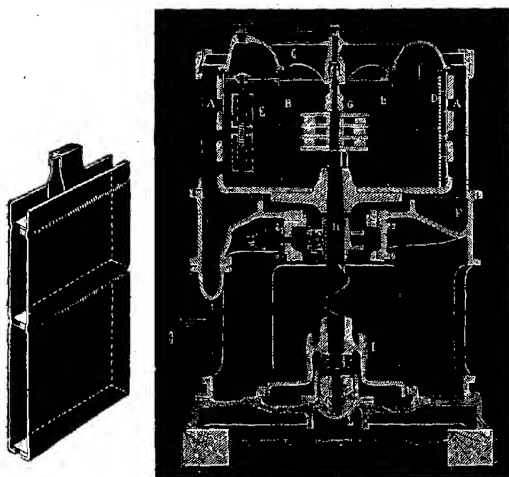
either by means of a rotating blade called the facing machine, or by the triangular instrument already described, so that all the loose, irregularly aggregated, and imperfectly crystallised sugar on the face of the loaf is removed. The cones are now ready to be knocked out of their moulds and subjected to further treatment, such as having their wet apices or noses cut off. They are knocked out of their moulds by a few blows as they lie horizontally. They are then stood on their base, the mould removed and the loaves left some time in the air covered by a paper hood to prevent them getting soiled. But the treatment varies with the dimensions and other characters of the sugar-loaves themselves. Those cones, which are over 16 lb. in weight, and known in commerce as lumps or titlers, have, as just stated, simply their apices truncated by a knife struck by a wooden mallet. But the apices of real sugar-loaves, in the proper acceptation of the term, are retained, or, rather, new apices are affixed to them by means of a conical cutter attached to a kind of turning-lathe. This tool is known in the trade as the "nosing machine". Nothing further requires to be done to the loaves except to dry them. The next step, therefore, whether in the case of loaves, lumps or titlers, consists in wrapping them in paper. The sugar-cones in their paper envelopes, so as to prevent them from getting dirty, are then deposited on trellised shelves in a room capable of holding about 4,000 loaves, called the stove, and heated by steam to about 54° C. (130° F.). The loaves are kept in the stove for a period of four to six days, after the expiry of which time they are equalised at face and apex, wrapped in paper, tied, and are then ready for sale.

All these operations take a long time and require an enormous space, as all the draining syrups are reboiled and are used in the manufacture of inferior quality sugars of the same form as the "firsts" sugars.

*Facilitating percolation and drainage of sugar-loaves by suction.*—When the drainage syrup issues colourless, the moulds are subjected to suction. The operation is conducted as follows. Connected with the air-pump are horizontal pipes furnished with conical caps with india-rubber fittings, on which the moulds are fixed air-tight with the apex downwards. A vacuum made in the pipe by a pump sucks the syrup, and removes the last dregs of the now useless cleansing liquor.

*Sugar tablets.*—There is another method of refining sugar, consisting in the manufacture of sugar tablets. In the case of a round sugar-loaf it must be cut into round tablets, perpendicular to the axis, and then broken into regular lumps for sale. All the lumps formed by the circumference are irregularly shaped and of less value. The question therefore was: why not make quadrangular tablets which would only produce regular cubes without waste? That is

the reason why, attempts have been made to change the classical form of the sugar-loaf and to replace it by regular tablets, which only require to be broken parallel with the sides to produce uniform cubes. The manufacture of sugar in tablet form has assumed enormous developments. In certain refineries attached to the French beet-sugar factories they only make this kind of sugar. The first stage of the refining process is the same. The change does not take place till after the boiling, when, instead of pouring the hot mass into moulds, it is poured into the ordinary rectangular moulds placed on a truck which is brought directly underneath the reheater to be filled. These moulds are then placed in turbines. They must



FIGS. 104, 105.—Sugar-tablet mould and turbine for cube sugar.

therefore be of such a shape as will allow this to be done easily. They consist of rectangular wrought-iron frames, eight inches across, and of the same height as the drum of the turbine. These frames are divided into compartments of about an inch thick by sheet-iron partitions which move on slides. To fill these frames, several are piled together, one on the top of another, keeping them firmly together, and the boiled mass is then run in. They are then left to cool, after which the frames are separated and firmly fixed in a turbine specially designed for the purpose (Fig. 105), which is turned for twenty minutes. The first molasses, or green syrup, flows away

by itself under the action of the centrifugal force. Then by means of a measuring vessel the necessary quantity of cleansing liquor is introduced to the centre of the turbine, to purify it. After another twenty minutes the liquor has penetrated the blocks of sugar. The revolutions are kept up for another twenty minutes, and the operation is finished. The frames are then dismantled, the tablets extracted and placed in the stove for about eight hours; finally, the tablets are broken by special machines and packed in boxes for sale. This process, more or less modified, improved and adapted by the different inventors<sup>1</sup> who have contributed to render it practicable, is a very rapid one, and yields excellent results. Nevertheless, the sugar-loaf yields more brilliant and more beautiful sugars, and will always preserve its value and prestige.

*Raw syrup or molasses.*—The residual green syrup or molasses remains to be treated or sold separately. In France, in the present state of the fiscal legislation of that country, molasses are no longer sold. They are again wrought up for sugar. A long time ago they were osmosed. Then they were treated with baryta, a process which was abandoned on account of the difficulties connected with the regeneration of the carbonate. They then fell back on strontia, using the same processes as in beet-sugar works. At the present time Steffen's process is again about to be used in French refineries, so as to abolish molasses as far as practicable. At the balancing of accounts or stocktaking the waste in manufacture is very small, much less than that allowed by the Legislature; that is what constitutes the bounty of the French sugar refiner.

*Manufacture of caramel.*—In addition to colouring alcoholic liquors, vinegar, etc., caramel is now used in photography. Common spirits are coloured by crude or caramelised molasses. Superior qualities of spirit by caramelised sugar, etc. The manufacture of caramel necessitates great care, otherwise the quality of the product may be injured.

*Manufacture of caramel from molasses.*—Sugar-cane molasses is alone used. It is run into a pan with half its volume of water; when the liquid boils it froths abundantly; a little broken up virgin wax is thrown into the pan to calm the emulsion. When the operation is finished, which is recognised by the mixture adhering to the spatula, it is withdrawn from the fire, cooled, and boiling water added gradually to dissolve the caramel. The pan is replaced on the fire and the mass stirred until it is quite homogeneous; it is filtered through a fine filter, and the cooled liquid bottled and a little alcohol added to preserve it.

*Caramel from sugar* is made in the same way. The sugar is run into a pan and heated with one-tenth of its weight of water, at first

<sup>1</sup> See Abridgment of Specifications for Sugar Patents for further details.

gently then more strongly. The sugar melts, and becomes coloured as it caramelises; when the colour is sufficiently accentuated the caramelisation is stopped, the pan is taken off the fire, and the remainder of the operation is the same as for molasses caramel.

*Caramel from glucose.*—Glucose can be caramelised in the same way as cane sugar. Generally glucose is caramelised in presence of alkalies, lime or soda, preferably carbonate of soda. Carbonate of soda in the proportion of 3 per cent. on the weight of the glucose is first dissolved in the caramel pan in double its weight of water, then the glucose is introduced. The process is then the same as in the case of sugar caramel. It is stirred, and when caramelised diluted with hot water, filtered and bottled. Glucose caramel prepared with an alkali is slightly acid, which is not the case when it is made from glucose alone. Tilting pans are made which tilt on a portable furnace, so that the diluted caramel may be filtered and bottled easily.

## SECTION IV.

### CHAPTER XII.

#### THE CHEMISTRY OF SUGARS—ANALYSIS OF COMMERCIAL SUGARS AND OF MERCHANDISE, ETC., CONTAINING SUGARS

**DEFINITION.**—Sugars are sweet substances soluble in water, and capable of being decomposed into carbonic acid, alcohol and some minor products by a peculiar chemical reaction induced by the action of a ferment, *e.g.*, yeast. This change of sugar into alcohol, etc., constitutes the alcoholic or vinous fermentation. All sugars belong to an important class of organic bodies, the *carbohydrates*, not that they contain water, but that they contain carbon, hydrogen and oxygen, and that the hydrogen and oxygen are present in the same proportion as in water. Sugars may be divided into three classes: 1. The *glucoses* or *monoses*—those analogous to grape sugar (glucose),  $C_6H_{12}O_6$ , molecular weight 180. 2. The *saccharoses* or *bioses*—those analogous to cane sugar (sucrose),  $C_{12}H_{22}O_{11}$ , molecular weight 342, sucrose, lactone, maltose. 3. The *trioses*—raffinose analogous with melitose, etc.,  $C_{18}H_{32}O_{16}$ , molecular weight 504.

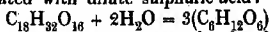
1. *Grape* sugar (glucose), the principal member of the first group, is the principal constituent of the juice of the grape and other sweet fruits. Honey also contains much grape sugar.

2. *Cane* sugar (sucrose) and *milk* sugar (lactose) are the chief sugars of the second group. Sucrose is got from the juice of (1) The sugar cane; (2) the sugar beet; (3) the sugar maple; (4) the sugar palms, and (5) is also present in most sweet fruits, *e.g.*, bananas, associated with sugars belonging to group 1. When purified the pure sucrose from each of these sources is identical. Milk sugar,  $C_{12}H_{22}O_{11} + H_2O$ , is present in the milk of all mammals, and is prepared by evaporating down the whey and letting the sugar crystallise out.

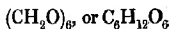
3. *Maltose* results from action of *diastase* on starch.—In addition to raffinose (p. 399) the *trioses* include such saccharine bodies as melitose extracted from Australian manna, and melezitose extracted by Berthelot from Braiçon manna secreted by the larch

## TECHNOLOGY OF SUGAR

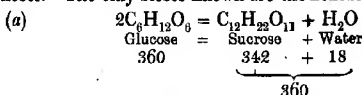
(Fr. *mélasse*). The trioses split up into three glucoses when their solutions are heated with dilute sulphuric acid:—



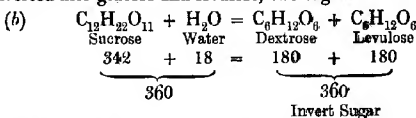
The monoses (glucoses) are compounds, the functions and behaviour of which are complex; they behave at the one time (*a*) as polyatomic alcohols, at other times (*b*) as aldehydes, or (*c*) as ketones. By hydrogenation they reproduce a polyatomic alcohol. The most important members are the hexamonomes or glucoses, the empirical formula of which is—



The bioses are produced by the combination of two monoses, with loss of water. They may be likened to the oxide ethers of the monoses. The only bioses known are the hexobioses or saccharoses—



If we boil cane sugar with a dilute acid it takes up water and is converted into glucose and levulose, two sugars of the first group—

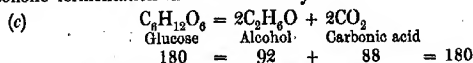


Milk sugar is acted on in the same way.

*Differentiation of the monoses from the bioses; difference in stability under action of reagents, ferments, etc.*—These two classes of sugars are principally differentiated from each other by their different susceptibility to alteration in their chemical constitution; the second group, i.e., the bioses or sucrose group, being more stable than the first or monose group (glucoses).

*Bioses do not directly ferment.*—The bioses, for instance, do not ferment directly; they are only capable of undergoing the alcoholic fermentation after having first fixed the elements of water, and thus become changed into sugars of the grape-sugar class, glucoses or monoses, and then the latter ferment:

*Direct fermentation and unstable nature of the monoses.*—On the other hand, the sugars of the grape-sugar class easily undergo alcoholic fermentation in contact with yeast—



Moreover, they begin to be decomposed when treated with caustic alkalis in the cold, and at 100° C. (212° F.) the reaction is complete. They reduce cupro-potassic solutions in the cold, or at least at 100° C.

The reagents which decompose monoses have little or no action on the bioses.—But caustic alkalies have little or no action on sugars belonging to the sucrose group even at 100° C. (212° F.), and at that temperature they only act feebly upon cupro-potassic solutions.

Sugar of the sucrose (cane sugar) class (bioses) crystallise readily, those of the monose (glucose) class with difficulty.—The two classes of sugars

TABLE LVIII.—DENSITY OF SOLUTIONS OF SUCROSE OF DIFFERENT STRENGTHS AT 17°·5 C.<sup>1</sup>

Per Cent.	Density.		Per Cent.	Density.		Per Cent.	Density.	
	Balling.	Niemann.		Balling.	Niemann.		Balling.	Niemann.
1	1·0040	1·0085	26	1·1106	1·1103	51	1·2385	1·2378
2	1·0080	1·0070	27	1·1153	1·1150	52	1·2441	1·2434
3	1·0120	1·0106	28	1·1200	1·1197	53	1·2479	1·2490
4	1·0160	1·0143	29	1·1247	1·1245	54	1·2553	1·2546
5	1·0200	1·0179	30	1·1295	1·1293	55	1·2610	1·2602
6	1·0240	1·0215	31	1·1343	1·1340	56	1·2667	1·2658
7	1·0281	1·0254	32	1·1391	1·1388	57	1·2725	1·2714
8	1·0322	1·0291	33	1·1440	1·1436	58	1·2783	1·2770
9	1·0363	1·0328	34	1·1490	1·1484	59	1·2841	1·2826
10	1·0404	1·0367	35	1·1540	1·1533	60	1·2900	1·2882
11	1·0446	1·0410	36	1·1590	1·1582	61	1·2959	1·2938
12	1·0488	1·0456	37	1·1641	1·1631	62	1·3019	1·2994
13	1·0530	1·0504	38	1·1692	1·1681	63	1·3079	1·3050
14	1·0572	1·0552	39	1·1743	1·1731	64	1·3139	1·3105
15	1·0614	1·0600	40	1·1794	1·1781	65	1·3190	1·3160
16	1·0657	1·0647	41	1·1846	1·1832	66	1·3260	1·3215
17	1·0700	1·0693	42	1·1898	1·1883	67	1·3321	1·3270
18	1·0744	1·0738	43	1·1951	1·1935	68	1·3383	1·3324
19	1·0788	1·0784	44	1·2004	1·1989	69	1·3445	1·3377
20	1·0832	1·0830	45	1·2057	1·2043	70	1·3507	1·3430
21	1·0877	1·0875	46	1·2111	1·2098	71	1·3570	1·3483
22	1·0922	1·0920	47	1·2165	1·2153	72	1·3633	1·3535
23	1·0967	1·0965	48	1·2219	1·2209	73	1·3696	1·3587
24	1·1013	1·1010	49	1·2274	1·2265	74	1·3760	1·3658
25	1·1059	1·1056	50	1·2329	1·2322			

are further differentiated by the readiness with which sucrose crystallises, whilst not only is grape sugar (*glucose*) difficultly and imperfectly crystallisable, and is usually obtained in a granular state, but it is much inferior in sweetness. The essential characteristic of the bioses of which cane sugar (*sucrose*) is the type is their splitting up under the action of dilute acids or of ferments into two

<sup>1</sup> Cp. Scheibler's Table VII., p. 12.



*glucoses (monoses)*, thus showing their intimate connection with the *glucosides* (reaction (b), p. 338).

TABLE LIX.—CORRECTIONS OF DEGREES BALLING FOR TEMPERATURES OTHER THAN 17°·5 C. (STAMMER SACHS).

Temperature Centigrade.	Degrees Balling of the Solution.												
	0	5	10	15	20	25	30	35	40	50	60	70	75
To be Added to the Observed Indication.													
Deg.													
0	0·27	0·34	0·41	0·52	0·62	0·72	0·82	0·92	0·98	1·11	1·22	1·25	1·29
5	0·23	0·30	0·37	0·44	0·52	0·59	0·65	0·72	0·75	0·80	0·88	0·91	0·94
10	0·20	0·27	0·29	0·33	0·36	0·39	0·42	0·45	0·48	0·50	0·54	0·58	0·61
11	0·18	0·23	0·26	0·28	0·31	0·34	0·36	0·39	0·41	0·43	0·47	0·50	0·53
12	0·16	0·20	0·22	0·24	0·26	0·29	0·31	0·33	0·34	0·36	0·40	0·42	0·46
13	0·14	0·18	0·19	0·21	0·22	0·24	0·26	0·27	0·28	0·29	0·33	0·35	0·39
14	0·12	0·15	0·16	0·17	0·18	0·19	0·21	0·22	0·22	0·23	0·26	0·28	0·32
15	0·09	0·11	0·12	0·14	0·14	0·15	0·16	0·17	0·17	0·17	0·19	0·21	0·25
16	0·06	0·07	0·08	0·09	0·10	0·10	0·11	0·12	0·12	0·12	0·14	0·16	0·18
17	0·02	0·02	0·03	0·03	0·03	0·04	0·04	0·04	0·04	0·04	0·05	0·05	0·06
To be Subtracted from Observed Indication.													
18	0·02	0·03	0·03	0·03	0·03	0·03	0·03	0·03	0·03	0·03	0·03	0·03	0·02
19	0·06	0·08	0·08	0·09	0·09	0·10	0·10	0·10	0·10	0·10	0·10	0·08	0·06
20	0·11	0·14	0·15	0·17	0·17	0·18	0·18	0·18	0·19	0·19	0·18	0·15	0·11
21	0·16	0·20	0·22	0·24	0·24	0·25	0·25	0·25	0·26	0·26	0·25	0·22	0·18
22	0·21	0·26	0·29	0·31	0·31	0·32	0·32	0·32	0·33	0·34	0·32	0·29	0·25
23	0·27	0·32	0·35	0·37	0·38	0·39	0·39	0·39	0·40	0·42	0·39	0·36	0·33
24	0·32	0·38	0·41	0·43	0·44	0·46	0·46	0·47	0·47	0·54	0·46	0·43	0·40
25	0·37	0·44	0·47	0·49	0·51	0·53	0·54	0·55	0·55	0·58	0·54	0·51	0·48
26	0·43	0·50	0·54	0·56	0·58	0·60	0·61	0·62	0·62	0·66	0·62	0·58	0·55
27	0·49	0·57	0·61	0·63	0·65	0·68	0·68	0·69	0·70	0·74	0·70	0·65	0·62
28	0·56	0·64	0·68	0·70	0·72	0·76	0·76	0·78	0·78	0·82	0·78	0·72	0·70
29	0·63	0·71	0·75	0·78	0·79	0·84	0·84	0·86	0·86	0·90	0·86	0·80	0·78
30	0·70	0·78	0·82	0·87	0·87	0·92	0·92	0·94	0·94	0·98	0·94	0·88	0·86
35	1·10	1·17	1·22	1·24	1·30	1·32	1·33	1·35	1·36	1·39	1·34	1·27	1·25
40	1·50	1·61	1·67	1·71	1·73	1·79	1·79	1·80	1·82	1·88	1·78	1·69	1·65
50	...	2·65	2·71	2·74	2·78	2·80	2·80	2·80	2·80	2·79	2·70	2·56	2·51
60	...	3·88	3·88	3·88	3·88	3·88	3·88	3·88	3·90	3·82	3·70	3·43	3·41
70	...	...	5·18	5·20	5·14	5·13	5·10	5·08	5·06	4·90	4·72	4·47	4·35
80	...	...	6·62	6·59	6·54	6·46	6·38	6·30	6·26	6·06	5·82	6·50	5·33

Having now given a brief chemical classification of sugars, it will be convenient to study them in the following order: I. Sucrose, II. Dextrose, III. Levulose, IV. Maltose, V. Lactose.

# THE CHEMISTRY OF SUGARS

341

TABLE LX.—TABLE EXHIBITING THE QUANTITY OF SUGAR, IN POUNDS AVOIR-  
DUPOIS, WHICH IS CONTAINED IN ONE GALLON OF SYRUP, AT SUCCESSIVE  
DEGREES OF DENSITY, AT 60° F. (URR).

Specific Gravity.	Lb. per Gallon.	Extract by Weight in 100.	Specific Gravity.	Lb. per Gallon.	Extract by Weight in 100.	Specific Gravity.	Lb. per Gallon.	Extract by Weight in 100.
1.000	0.0000	.0000	1.052	1.3472	.1278	1.103	2.7188	.2421
1.001	0.0255	.0026	1.053	1.3788	.1901	1.104	2.7446	.2443
1.002	0.0510	.0051	1.054	1.4004	.1925	1.105	2.7704	.2464
1.003	0.0765	.0077	1.055	1.4270	.1948	1.106	2.7961	.2486
1.004	0.1020	.0102	1.056	1.4536	.1972	1.107	2.8227	.2507
1.005	0.1275	.0128	1.057	1.4802	.1995	1.108	2.8485	.2529
1.006	0.1530	.0153	1.058	1.5068	.1418	1.109	2.8740	.2550
1.007	0.1785	.0179	1.059	1.5334	.1441	1.110	2.9001	.2571
1.008	0.2040	.0204	1.060	1.5600	.1464	1.111	2.9268	.2593
1.009	0.2295	.0230	1.061	1.5870	.1487	1.112	2.9522	.2614
1.010	0.2550	.0255	1.062	1.6142	.1510	1.113	2.9780	.2635
1.011	0.2805	.0280	1.063	1.6414	.1533	1.114	3.0045	.2656
1.012	0.3060	.0306	1.064	1.6688	.1556	1.115	3.0304	.2677
1.013	0.3315	.0331	1.065	1.6959	.1579	1.116	3.0563	.2698
1.014	0.3570	.0356	1.066	1.7228	.1602	1.117	3.0821	.2719
1.015	0.3825	.0381	1.067	1.7496	.1625	1.118	3.1080	.2740
1.016	0.4180	.0406	1.068	1.7764	.1647	1.119	3.1343	.2761
1.017	0.4335	.0431	1.069	1.8033	.1670	1.120	3.1610	.2782
1.018	0.4590	.0456	1.070	1.8300	.1693	1.121	3.1871	.2803
1.019	0.4845	.0481	1.071	1.8571	.1716	1.122	3.2130	.2824
1.020	0.5100	.0506	1.072	1.8843	.1738	1.123	3.2399	.2845
1.021	0.5355	.0531	1.073	1.9116	.1761	1.124	3.2658	.2865
1.022	0.5609	.0555	1.074	1.9385	.1783	1.125	3.2916	.2886
1.023	0.5853	.0580	1.075	1.9653	.1806	1.126	3.3174	.2907
1.024	0.6104	.0605	1.076	1.9928	.1828	1.127	3.3431	.2927
1.025	0.6355	.0629	1.077	2.0197	.1851	1.128	3.3690	.2948
1.026	0.6606	.0654	1.078	2.0465	.1873	1.129	3.3949	.2969
1.027	0.6857	.0679	1.079	2.0734	.1896	1.130	3.4211	.2989
1.028	0.7108	.0703	1.080	2.1006	.1918	1.131	3.4490	.3010
1.029	0.7359	.0727	1.081	2.1275	.1941	1.132	3.4769	.3030
1.030	0.7610	.0752	1.082	2.1548	.1963	1.133	3.5048	.3051
1.031	0.7861	.0776	1.083	2.1811	.1985	1.134	3.5326	.3071
1.032	0.8112	.0800	1.084	2.2080	.2007	1.135	3.5605	.3092
1.033	0.8363	.0825	1.085	2.2359	.2029	1.136	3.5882	.3112
1.034	0.8614	.0849	1.086	2.2627	.2051	1.137	3.6160	.3132
1.035	0.8866	.0873	1.087	2.2894	.2073	1.138	3.6437	.3153
1.036	0.9119	.0897	1.088	2.3161	.2095	1.139	3.6716	.3173
1.037	0.9449	.0921	1.089	2.3438	.2117	1.140	3.7000	.3193
1.038	0.9768	.0945	1.090	2.3710	.2139	1.141	3.7281	.3214
1.039	1.0090	.0969	1.091	2.3987	.2161	1.142	3.7562	.3234
1.040	1.0400	.0993	1.092	2.4256	.2183	1.143	3.7840	.3254
1.041	1.0658	.1017	1.093	2.4524	.2205	1.144	3.8119	.3274
1.042	1.0906	.1041	1.094	2.4792	.2227	1.145	3.8398	.3294
1.043	1.1159	.1065	1.095	2.5061	.2249	1.146	3.8677	.3314
1.044	1.1412	.1089	1.096	2.5329	.2270	1.147	3.8955	.3334
1.045	1.1665	.1113	1.097	2.5598	.2292	1.148	3.9235	.3354
1.046	1.1918	.1136	1.098	2.5866	.2314	1.149	3.9516	.3374
1.047	1.2171	.1160	1.099	2.6130	.2335	1.150	3.9801	.3394
1.048	1.2424	.1184	1.100	2.6404	.2357	1.151	4.0070	...
1.049	1.2687	.1207	1.101	2.6668	.2378	1.152	4.0342	...
1.050	1.2940	.1231	1.102	2.6921	.2400	1.153	4.0611	...
1.051	1.3206	.1254						

TABLE LX.—Continued.

Specific Gravity.	Lb. per Gallon.	Specific Gravity.	Lb. per Gallon.	Specific Gravity.	Lb. per Gallon.	Specific Gravity.	Lb. per Gallon.	Specific Gravity.	Lb. per Gallon.
1.154	4.0880	1.184	4.8802	1.214	5.6651	1.243	6.4650	1.272	7.2902
1.155	4.1148	1.185	4.9051	1.215	5.6942	1.244	6.4902	1.273	7.3204
1.156	4.1319	1.186	4.9300	1.216	5.7233	1.245	6.5153	1.274	7.3506
1.157	4.1588	1.187	4.9552	1.217	5.7522	1.246	6.5402	1.275	7.3807
1.158	4.1857	1.188	4.9803	1.218	5.7814	1.247	6.5651	1.276	7.4109
1.159	4.2128	1.189	5.0054	1.219	5.8108	1.248	6.5903	1.277	7.4409
1.160	4.2502	1.190	5.0304	1.220	5.8401	1.249	6.6152	1.278	7.4708
1.161	4.2771	1.191	5.0563	1.221	5.8680	1.250	6.6402	1.279	7.5007
1.162	4.3040	1.192	5.0822	1.222	5.8962	1.251	6.6681	1.280	7.5307
1.163	4.3309	1.193	5.1080	1.223	5.9242	1.252	6.6960	1.281	7.5600
1.164	4.3578	1.194	5.1341	1.224	5.9523	1.253	6.7240	1.282	7.5891
1.165	4.3847	1.195	5.1602	1.225	5.9801	1.254	6.7521	1.283	7.6180
1.166	4.4115	1.196	5.1868	1.226	6.0081	1.255	6.7800	1.284	7.6469
1.167	4.4383	1.197	5.2124	1.227	6.0361	1.256	6.8081	1.285	7.6758
1.168	4.4652	1.198	5.2381	1.228	6.0642	1.257	6.8362	1.286	7.7048
1.169	4.4923	1.199	5.2639	1.229	6.0925	1.258	6.8643	1.287	7.7331
1.170	4.5201	1.200	5.2901	1.230	6.1205	1.259	6.8921	1.288	7.7620
1.171	4.5460	1.201	5.3160	1.231	6.1474	1.260	6.9201	1.289	7.7910
1.172	4.5722	1.202	5.3422	1.232	6.1743	1.261	6.9510	1.290	7.8201
1.173	4.5983	1.203	5.3681	1.233	6.2012	1.262	6.9822	1.291	7.8482
1.174	4.6242	1.204	5.3941	1.234	6.2280	1.263	7.0133	1.292	7.8763
1.175	4.6505	1.205	5.4203	1.235	6.2551	1.264	7.0444	1.293	7.9042
1.176	4.6764	1.206	5.4462	1.236	6.2822	1.265	7.0751	1.294	7.9321
1.177	4.7023	1.207	5.4720	1.237	6.3093	1.266	7.1060	1.295	7.9600
1.178	4.7281	1.208	5.4979	1.238	6.3362	1.267	7.1369	1.296	7.9879
1.179	4.7539	1.209	5.5239	1.239	6.3631	1.268	7.1678	1.297	8.0158
1.180	4.7802	1.210	5.5506	1.240	6.3903	1.269	7.1988	1.298	8.0448
1.181	4.8051	1.211	5.5786	1.241	6.4152	1.270	7.2300	1.299	8.0719
1.182	4.8303	1.212	5.6071	1.242	6.4401	1.271	7.2601	1.300	8.1001
1.183	4.8554	1.213	5.6360						

I. *Sucrose (cane sugar).*—(a) *Properties.*—Sugar crystallises in hard, anhydrous, oblique, rhomboidal prisms of density 1.606 (sugar candy) with hemihedral facets, and phosphorescing when rubbed or crushed in the dark.

(b) *Solubility in water.*—Sugar dissolves in one-third of its weight of cold water to a thick and viscous solution known as simple syrup. A solution of 100 parts of sugar in 50 parts of water (i.e., 20 lb. sugar to 1 gallon water) shows a density of 1.345 at 15° C., and marks 37° by Baumé (69° Tw.). This solution boils at 105° C. (221° F.). The pounds of sugar in a gallon of syrup may be approximately got by multiplying the decimal part of the gravity by 26 (Üre). The aqueous solution deviates the plane of polarisation to the right (p. 362), and the rotary power does not diminish on keeping the solution, not even on boiling (Bechamp).

(c) *Solubility in alcohol.*—Sugar is insoluble in ether and in cold absolute alcohol. Boiling absolute alcohol dissolves about  $1\frac{1}{2}$  per cent. Boiling ordinary alcohol dissolves more. Sugar is deposited from its alcoholic solution on cooling as small but distinct crystals,

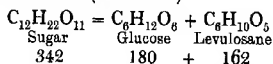
TABLE LXI.—SOLUBILITY OF SUCROSE IN MIXTURES OF ALCOHOL AND WATER (SCHEIBLER).

Percentage of Alcohol.	At 0° C.		At 14° C.		At 40° C.
	Density at 17°·5 C.	Sugar per Cent.	Density at 17°·5 C.	Sugar per Cent.	Sugar per Cent.
		Grammes.		Grammes.	
0	1·3248	85·5	1·3258	87·5	105·2
10	1·2991	80·7	1·3000	81·5	95·4
20	1·2860	74·2	1·2662	74·5	90·0
30	1·2293	65·5	1·2327	67·9	82·2
40	1·1823	56·7	1·1848	58·0	74·9
50	1·1294	45·9	1·1305	47·1	63·4
60	1·0500	32·9	1·0582	33·9	49·9
70	0·9721	18·2	0·9746	18·8	31·4
80	0·8931	6·4	0·8953	6·6	13·3
90	0·8369	0·7	0·8376	0·9	2·3
97·4	0·8062	0·08	0·8062	0·36	0·5

(d) *Action of reagents.*—Sucrose does not ferment directly, but in the presence of the yeast ferment and of the peculiar ferments contained in most saccharine fruits it fixes water and is converted into a mixture of dextrose and levulose known as *invert sugar*, capable of undergoing fermentation (see equations (b) and (c), p. 338). This transformation is also effected when sugar is boiled for a long time in water. Hence the precaution which is taken in the manufacture of sugar to evaporate the clarified juices at a low temperature. Under the action of *dilute acids* (equation (b), p. 338), sucrose is likewise changed into invert sugar. The change takes place slowly in the cold, rapidly on boiling. Dilute sulphuric acid is more effectual than other, and more especially organic, acids. The original solution deviates the plane of polarisation to the right (p. 362). After treatment with dilute acids and cooling, the liquid deviates to the left (p. 362), for the rotary power of levulose to the left is greater than that of glucose to the right. Organic acids produce the same effect on boiling, but at the ordinary temperature their action is extremely slow—which explains the coexistence of sucrose and of acetic, malic and tartaric acids in certain fruit sugars. When sucrose is subjected to *prolonged ebullition* with dilute sulphuric or hydrochloric acids the solution eventually becomes brown, and ulmic substances are

formed. Organic acids of the fatty series ( $C^nH^{2n+1}$ )COHO, such as acetic, butyric or stearic acids, combine with sugar at  $120^\circ$  C., forming neutral bodies analogous to oils and fats. *Boiling glacial acetic acid* transforms cane sugar in a few minutes into an acetic ether. *Concentrated sulphuric acid* carbonises sucrose rapidly. The action is especially rapid with hot acid, generates heat, and produces an evolution of sulphurous acid gas, with much intumescence of a bulky black carbonaceous slime. *Concentrated nitric acid* converts sugar into oxalic acid. A mixture of bichromate of potash, peroxide of manganese and sulphuric acid energetically oxidise sucrose with production of formic acid. By adding powdered sugar to a mixture of nitric and sulphuric acids, cooled to  $2^\circ$  C., a nitrated product is obtained, which would appear to consist of tetranitric saccharose formula. It forms an amorphous mass which detonates on concussion. Dry chlorine does not act on dry sucrose. Chlorine attacks sucrose syrup at a temperature of  $100^\circ$  C., and forms hydrochloric acid and brown compounds. Certain chlorides, such as antimonie and stannic chlorides, impart a brown coloration to sucrose.

(e) *Action of heat*.—Sucrose melts at  $160^\circ$  C. into a thick transparent liquid, which on cooling is transformed into an amorphous vitreous mass (*barley sugar*); but gradually, in consequence of molecular change, the mass again becomes crystalline. When sucrose is kept for a long time at  $160^\circ$  to  $161^\circ$  C. it splits up into *glucose* and *levulosane* (saccharide)—



When this mixture of glucose and levulosane is dissolved in water and fermented the glucose first disappears, and the levulosane remains behind, and may be isolated by evaporating the solution and heating the residue to  $170^\circ$  C.

(f) *Caramel*.—When sugar is heated from  $190^\circ$  to  $220^\circ$  C. it continuously loses water, and is converted into a brown, amorphous, bitter substance, soluble in water, known under the name of caramel. (The manufacture and uses of commercial caramel from cane or beet sugar, glucose, molasses, etc., is described on pp. 335-6.)

Sucrose exposed for seven hours to a temperature of  $300^\circ$  F. only lost 0.6 per cent. of its weight, but its properties were permanently altered (Prout). When sucrose is heated in an oil bath to  $410^\circ$  to  $420^\circ$  F. it becomes darker coloured without evolving any gaseous products, but giving off water slightly tainted with acetic acid and oil; when this ceases, the residue is black and brilliant, but perfectly soluble in water, yielding a dark solution (Peligot). When sucrose is subjected to dry distillation it is decomposed, and completely

carbonised with formation of carbonic oxide, carbonic acid, marsh gas, acetic acid, aldehyde, acetone, liquid hydrocarbides,<sup>1</sup> and empyreumatic products. The products are partially formed when sugar is heated in a platinum crucible. It intumesces, gives off a very peculiar odour, the smell of burnt sugar, takes flame and leaves a bulky residue of very light carbon. Dr. E. J. Mills distilled cane sugar in a glass flask—25 grammes; time taken, twelve hours. The corrected results were—

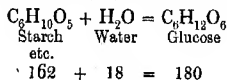
$$\begin{array}{rcccc} \text{C}_{12}\text{H}_{22}\text{O}_{11} & = & 9\text{C} & + & \text{C}_2\text{H}_2\text{O} & + & 10\text{H}_2\text{O} \\ 342 & & 108 & + & 54 & & 180 = 342 \end{array}$$

	Fixed Carbon.	Gas and Tar.	Water (Organic).
Calculated . . . .	31.6	15.8	52.6
Found . . . . .	31.5	17.7	50.8

Sugar furnishes 2.4 of acetate when thus distilled, and very little tar. The gas probably amounted to 17.5 per cent. High temperature cane-sugar coke contains 95 per cent. of carbon and 1.2 per cent. of hydrogen. Fischer and Laycock found the distillate to contain propylaldehyde and dimethylfurfuran.

(g) *Sucrates and Sucro-Carbonates*.—See pp. 73-5 and 225-230.

II. *Dextrose*.—(a) *History*.—Lowitz first differentiated in 1792 between sucrose and the crystallisable portion of honey. Proust determined, in 1802, the peculiar nature of grape sugar, which was afterwards found in a large number of saccharine fruits, such as figs, prunes, on the surface of which it forms, on drying, the well-known efflorescences. In addition to glucose, the greater number of saccharine fruits contain an equal amount of levulose, whilst some also contain sucrose (p. 343). The mixture of glucose and levulose present in fruits constitutes invert sugar. The saccharine substance present in diabetic urine consists of glucose, which is also present sometimes in a normal state of health in several other animal fluids. Kirchoff discovered, in 1811, that glucose could be prepared artificially by the action of dilute sulphuric acid on starch. In 1819 Braconnot, in a similar manner, transformed cellulose into glucose—



Again, it has been determined (pp. 349-50) that the saccharine substance capable of fermentation which may be separated from certain glucosides, such as amygdalin, salicin, tannic acid, is crystallisable and identical with glucose.

<sup>1</sup>The term *hydrocarbides* is used advisedly. "Hydrocarbon" as used for a compound of carbon and hydrogen does not harmonise with metallic "carbides".

(b) *Manufacture*.—Glucose may be manufactured industrially by utilising the transformation of farina and starch into glucose in presence of dilute sulphuric acid, accompanied by the application of heat. Three hundred and thirty gallons of water, to which 308 lb. of sulphuric acid have been added, are run into a capacious vat capable of holding 35,000 gallons, and steam is turned on through a copper coil pierced with holes in connection with a high-pressure boiler. Jets of superheated steam issue from the holes in the pipe. The starch, diluted with about its own weight of water, is run into the vessel by successive portions; as much as 10 tons of starch can be saccharified in this way in a single operation. The liquid having been kept boiling, saccharification is complete in one-half hour, a point, however, which can be ascertained by testing the solution with iodine; when tincture of iodine no longer colours the liquid violet the operation is finished. Nothing but glucose remains and a little achro dextrine. The operation may also be conducted in copper autoclaves of 550-660 gallons capacity, under a pressure of 3 to 4 atmospheres. The acidulated water is first run into the autoclave, then the starch beat up in water. The sulphuric acid is saturated by powdered chalk, and the whole allowed to stand; sulphate of lime is deposited; the liquid is run off, passed through animal charcoal filters analogous to those used in sugar houses. The liquid is concentrated in vacuum pans heated by steam until it shows 30° Baumé (sp. gr. 1·2569) (27° B., sp. gr. 1·2254, when it is boiling), and the syrup thus obtained is sold to brewers and the makers of spiced bread. A whiter syrup, more concentrated than the latter, termed *imponderable syrup* because its viscosity is such that a hydrometer will not sink in it, is sold to confectioners, for the making of fruit comfits, syrups, sweets, etc.

The syrup, concentrated to 30° B., and allowed to stand in casks for eight to ten days, deposits small mammillary crystals of glucose; the mother-liquid is decanted, and *granulated glucose* is obtained on drying. Finally, when the syrup is concentrated to 41° B. (sp. gr. 1·3876) it is gradually converted, on cooling, into a white hard mass called massive glucose or massive starch sugar (see Table LXVI, p. 366).

(c) *Extraction of glucose from honey*.—Glucose may be extracted from honey by beating up the latter with cold alcohol, which dissolves the uncrystallisable sugar. The liquid is decanted, and the solid strongly pressed between folds of filter paper; it is again beaten up with alcohol and pressed once more. The residue is dissolved in water, decolorised by animal charcoal, and set aside to crystallise.

(d) *Extraction of glucose from diabetic urine*.—In order to extract glucose from diabetic urine, the latter is evaporated on the water bath, or better, in an oven; a little alcohol is added, and it is afterwards left to crystallise. The crystalline mass is washed with cold alcohol and purified by several crystallisations in hot water, with the

addition of animal charcoal. Instead of dissolving the glucose in cold water, it may be dissolved in hot 90 per cent. alcohol. It crystallises more easily from alcohol. When urine is not very rich in glucose, it deposits, in addition to crystals of the latter, crystals consisting of a combination of glucose or common salt, or even these alone.

(e) *Properties*.—Glucose is generally met with as mammillary white opaque crystals, cauliflower like agglomeration containing two equivalents of water of crystallisation. They are permanent in the air, but soften at 60° C., melt in the water bath, and lose their water of crystallisation at 100° C., or even in a current of air at 80° C. Glucose is deposited from its alcoholic solution in anhydrous needles, which do not melt until 196° C. Glucose is three times less soluble in water than cane sugar, and its solution of equal strength is three times less sweet. One part of glucose dissolves in 1·2 of water at 17° C. The solution readily ferments (c), (p. 338). Glucose dissolves very easily in dilute alcohol, but is less soluble in absolute alcohol.

TABLE LXII.—(1) DENSITY OF AQUEOUS SOLUTIONS OF GLUCOSE (SALOMON), AND (2) SOLUBILITY OF GLUCOSE AT 64° F. IN ALCOHOL OF VARIOUS STRENGTHS (ARTHON).

Density of Aqueous Solutions of Glucose.						Solubility Per Cent. in Alcohol.		
%	Density.	%	Density.	%	Density.	Density of Alcohol.	Initial.	Permanent.
5	1·0192	25	1·0946	45	1·1680	0·837	1·95	1·94
10	1·0381	30	1·1180	50	1·1863	0·880	9·30	8·10
15	1·0571	35	1·1310	55	1·2040	0·910	17·74	16·00
20	1·0762	40	1·1494	50	1·2218	0·950	36·45	32·50

(f) *Action of heat on glucose*.—When glucose is heated to about 170° C. it melts, loses water, and is converted into glucosane—



Glucosane has not yet been obtained in a pure state. It does not ferment directly, but ebullition with acids changes it into fermentescible glucose. Glucose or glucosane caramelize when heated to high temperatures. The manufacture of caramel from glucose is described on p 336.

(g) *Action of acids*.—Glucose is not coloured when treated in the cold by concentrated sulphuric acid; it combines therewith, forming a sulphoglucosic acid analogous to sulphosaccharic acid. But if the temperature rises the glucose is charred with evolution of sulphurous acid. Dilute boiling nitric acid converts glucose into oxalic acid.

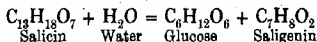
(h) *Action of bases*.—Alkaline bases decompose glucose slowly at first, rapidly on boiling. If a solution of caustic potash, e.g., be



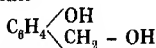
added to a solution of glucose, the liquid turns yellow as soon as heat is applied, afterwards turning a very deep brown. The same coloration is produced when glucose is heated with caustic baryta or caustic lime. This reaction is so delicate as to detect glucose when used as an adulterant of sucrose in commercial sugars; glucose combines with the alkaline earths and lead oxide to form crystalline products. It combines with caustic baryta to form a *glucosate of baryta* ( $4C_6H_{12}O_6, 3BaO + 4H_2O$ ), obtained as a white crystalline powder when an alcoholic solution of glucose is mixed with a solution of barium hydrate in weak alcohol. A basic glucosate of lead ( $C_6H_{12}O_6, 3PbO + 2H_2O$ ) is precipitated when ammonia is added to a mixed solution of glucose and lead acetate. When slaked lime is dissolved in a solution of glucose and the solution mixed with alcohol a white precipitate of glucosate of lime is formed.

(i) The reducing action of glucose is discussed pp. 364 *et seq.*

(j) *Glucosic ethers*.—Glucose behaves with acids like an alcohol; forms with acids, with elimination of 1, 2, 3, 4, or even 5 molecules of  $H_2O$ , true ethers. These ethers, the glucosides of Berthelot, are obtained by heating glucose in sealed tubes with an acid, but mineral acids exert a destructive action on glucose at temperatures between  $100^\circ$  and  $120^\circ C.$ , the temperatures at which these reactions take place. Glucosides, therefore, are not easily prepared, except those from organic acids (tartaric, acetic and stearic acids). By dissolving glucose in fuming nitric acid a pentanitric glucoside is obtained, thus establishing its pentatomic alcoholic nature. All glucosides split up in presence of dilute acids into glucose and the producing acid. Some of the glucosides of organic acids exist in acid fruits. Many neutral crystalline principles are met with in plants which split up like the above-mentioned glucosides into a glucose capable of fermentation; but one of the products is an alcohol or a phenylic alcohol, *e.g.*, salicin, the bitter principle of different species of elm, poplar, esculin contained in the bark of the purging nut tree, coniferin extracted from conifers, and which on oxidation with chromic acid mixture yields vanillin (see composition of beet juice). These neutral glucosides may be regarded as the result of the union of glucose with an alcohol, with elimination of water—



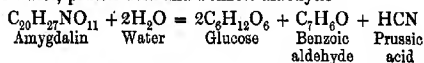
Saligenin is a phenylic alcohol—



The saccharoses (bioses) themselves, although their synthesis cannot be effected from two glucoses, also belong to the same class, by the manner in which they split up (see p. 338).

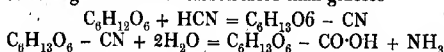
Moreover, glucosides are known which split up by absorbing the elements of water into a glucose, an acid, an alcohol or an aldehyde.

Such is *amygdalin*, contained in bitter almonds and the almonds of a number of stone fruits, and which yields, by decomposition in presence of water, prussic acid and benzoic aldehyde—

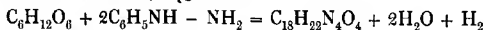


The above is the reaction by which the essential oil of bitter almonds is produced from amygdalin.

(k) *Osazones*.—Like aldehydes, glucose unites with prussic acid HCN to form a nitrile which saponified by KHO yields a monobasic acid containing one atom of carbon more than glucose—



It also reacts on two molecules of phenyl hydrazin  $\text{H}_5\text{N} - \text{NHC}_6\text{H}_5$  in acetic solution to form a crystalline compound *osazone*, with elimination of water and hydrogen thus—



III. *Levulose* (*fructose*, *fruit sugar*) ( $\text{C}_6\text{H}_{12}\text{O}_6$ ).—(a) *Preparation*.—Levulose—often termed uncrystallisable fruit sugar—forms the uncrystallisable portion of the saccharine matter of certain fruits (raisins, cherries, gooseberries), in which it is present along with glucose, and generally to the same extent, just as if they resulted from the inversion of sucrose by the action of acids (b), (p. 338). It also forms the uncrystallisable portion of honey. By heating sucrose with dilute acids it is changed into *invert sugar*, a mixture of equal weights of dextrose and levulose. Levulose in a pure state is obtained very rapidly by saccharifying inulin with dilute acids. (Inulin is a substance isomeric with starch, contained in the roots of elecampane, dahlia, colchicum and Jerusalem artichoke.) Levulose may be isolated from *invert sugar* by mixing 10 grammes of the latter with 6 grammes of slaked lime. The mass, which is at first liquid, becomes pasty on stirring. It then contains liquid glucosate of lime and solid levulosate of lime. It is put into a cloth and then subjected to pressure. The solid part remaining behind is the calcareous salt of levulose, all the glucose having been pressed out as glucosate of lime. The solid calcareous salt is dissolved in water and decomposed by a current of carbonic acid. The filtered and evaporated solution yields *pure levulose*, which may also be prepared as follows: Into a 50 c.c. *Erlenmeyer* flask put 50 c.c. water and 5 c.c. HCl, of density to give 0.01 to 0.02 per cent. HCl to weight of inulin. (For 200 grammes inulin of 0.25 per cent. ash content, 5 c.c. normal HCl; for higher ash content, more.) The 200 grammes of inulin, in fine powder, are then added and well shaken and heated in boiling water (not in bath). After thirty minutes, from time mass is evenly heated, the maximum of the inversion is reached, the flask is removed from the

boiling water. The syrup, carrying some granules of inverted inulin, is poured into 1 litre of absolute alcohol, a little blood char added, and allowed to stand for twenty-four hours, and filtered. A few crystals of fruit sugar added to the filtrate cause a copious crystallisation of pure levulose.

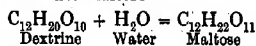
A better yield is got by evaporating the alcoholic filtrate to a thick syrup and placing over  $H_2SO_4$  in dessicator. A crop of crystals is thus obtained, which resemble dextrose got in similar conditions. The crystal masses thus obtained are dissolved in three to four parts absolute alcohol, allowed to stand twelve hours, and the clear solution poured off. A few crystals of fruit sugar and rubbing with a glass rod cause the crystallisation of the levulose to appear at once.

(b) *Solubility*.—Levulose when pure is an uncrystallisable syrup, with a much sweeter taste than glucose syrup. It dissolves very readily in water and in ordinary alcohol, but not so easily in absolute alcohol.

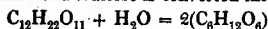
(c) *Rotary power*.—It is levo-rotary, and its power is  $-106^\circ$  at  $15^\circ C$ , but it varies greatly with the temperature: thus at  $90^\circ C$  it is decreased by one-half, becoming  $-53^\circ$ . But glucose having a dextro-rotary power which does not vary with the temperature, the variations in the rotary power of levulose ought to be still present in invert sugar, which is a mixture of equal parts of glucose and levulose. As a matter of fact, inverted sugar whose rotary power is  $-25^\circ$  at  $15^\circ C$  is only half as much at  $52^\circ C$ , is nil at  $90^\circ C$ , and changes its sign above that temperature.

(d) *Action of heat*.—Levulose begins to decompose above  $100^\circ C$ , and gives the same products of decomposition as glucose. With lime it forms an *insoluble* compound,  $2C_6H_{12}O_6, 3CaO$ . Although levulose decomposes much more readily under the influence of acids or of heat, yet it resists the action of alkalies or ferments much better. Its greater resistance to the action of ferments has been utilised in its preparation. If during the course of fermentation the rotary power of the fermenting liquid be taken from time to time, it will be found, at the end of a certain time, that the deviation to the left attains its maximum and then diminishes; on arresting the fermentation at this point the glucose is destroyed, and the liquid only contains levulose.

IV. *Maltose*.—Maltose is produced when amylaceous bodies are heated at  $60^\circ C$  with water and germinated barley (malt). Under the influence of the *diastase* the amylaceous matter is transformed into dextrine and then into maltose—

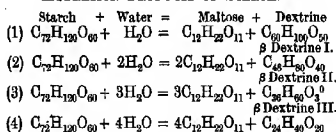


Crystallised maltose has the formula  $C_{12}H_{22}O_{11} + H_2O$ . Heated with dilute sulphuric acid maltose is converted into glucose—



Maltose is dextro-rotary like glucose, but to a far greater extent than ordinary glucose. It reduces cupro-potassic solutions, although not so easily as the glucoses. It ferments under the same conditions as cane sugar. Maltose sugar was first discovered by *Dubrunfaut*, but it was forgotten until *O'Sullivan* of Burton-on-Trent again studied the action of albuminoid ferments, such as are to be found in malt upon starch. The following table represents the reaction:—

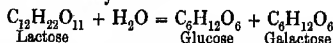
## HYDRATION PRODUCTS OF STARCH.



The first action is to add one molecule of water, which is effected by the action of albuminoid ferments. The same action is also brought about by the saliva of man, because that also contains an albuminoid ferment called *ptyaline*. In malt there is a similar ferment, *diastase*. When the first molecule of water is added it causes a lesion of the complex starch molecule into the distinct product maltose sugar ( $\text{C}_{12}\text{H}_{22}\text{O}_{11}$ ) (the isomer of cane sugar and five molecules of dextrine— $\text{C}_{12}\text{H}_{20}\text{O}_{10}$ ). The next stage is to add two molecules of water; then a further lesion is brought about by the ferment. In the last stage four molecules of water are added, and thus four molecules of maltose sugar and two of dextrine are produced. *Brown* and *Heron* and *Gruber* and *Musculin* give some other hydration products in addition to those given by *O'Sullivan*. Although it presents many analogies with cane sugar, yet maltose may be differentiated from the former by the fact that it is precipitated from its aqueous solution by alcohol, whilst cane sugar is not precipitated under similar conditions. Alcohol only dissolves maltose in minute proportions. Maltose also differs from cane sugar in the following respects. The formula is the same, that is, the composition of 100 parts is the same, and the molecular weight is perhaps the same, but the properties that distinguish maltose are the following. Cane sugar rotates the plane of polarised light to the right, maltose rotates the ray also to the right, but about twice as far. It is much more dextro-rotatory than cane-sugar and glucose, although less so than starch. *Fehling's* solution has no action on cane sugar, except, however, after a time, when the alkali begins to break up the sugar. It has no immediate action; but on maltose sugar *Fehling's* solution produces an abundant and immediate reaction on boiling; still, the amount of reduction of the protoxide of copper to suboxide is not so great as in the case of dextrose and levulose sugars (*Graham*).

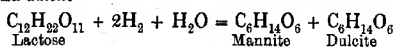
*V. Lactose or milk sugar* ( $\text{C}_{12}\text{H}_{22}\text{O}_{11} + \text{H}_2\text{O}$ ).—This substance was

first described by *Bartholetti* (*Encyclopædia Hermetico-dogmatica*, Bologna, 1619, "Manna, seu nitrum seri lactis"). But the person who contributed to make sugar of milk generally known was *Ludovico Festi*, who gave it out as an invention of his own, and sold it as a remedy in gout and other diseases. He was a physician in Venice, where he died in 1707. After his death *Valisneri* published the process which Festi employed in extracting this sugar from whey. Lactose or milk sugar can only be obtained by evaporating the whey obtained as a liquid by-product in cheese-making. It is said to have at one time been largely manufactured as an article of food in Switzerland. It may be prepared by coagulating the milk by a small quantity of sulphuric acid. The whey, filtered, evaporated, and the syrupy solution allowed to stand in an open place, deposits small but very hard crystals, which are purified by fresh crystallisation and bleaching by animal charcoal. It is met with in commerce in cylindrical pieces consisting of an agglomeration of crystals around a wooden stick serving as an axis. Milk sugar crystallises in right rhomboidal prisms terminating in octahedral points. *Density*. —1.53. It is hard, transparent, and gritty between the teeth. Its taste is but very slightly sweet. On keeping it becomes covered with moulds. It dissolves in six parts of cold water with evolution of heat and in two and a half parts of boiling water. Cold alcohol and ether do not dissolve lactose. Alcohol therefore precipitates lactose from its aqueous solution. Crystals of lactose dried at 100° C. have the formula  $C_{12}H_{22}O_{11} + H_2O$ . At 150° C. they lose their water of crystallisation, and have then the same centesimal composition as cane sugar. At this temperature the crystals begin to decompose. The dehydrated crystals become brown without melting at 160° C.; at 175° C. they change into *lactocaramel* ( $C_{12}H_{20}O_{10}$ ), an amorphous brown tasteless substance. Milk sugar does not melt until 203°·5 C. A solution of milk sugar is dextro-rotary. Its rotary power referred to the formula  $C_{12}H_{22}O_{11}$  is equal to 59°·3. It is greater by ·38 in recent solutions, but soon diminishes to the constant term. Like glucose, it reduces cupro-potassic solutions, even in the cold. Its reducing power is less than glucose, its cupreous precipitate being only one-tenth that of the latter, other things being equal. When lactose is boiled with dilute mineral acid it splits up into two glucoses, ordinary glucose and *galactose*, which is also dextro-rotary—



Lactose is carbonised at 100° C. under the action of fuming hydrochloric acid and of concentrated sulphuric acid. Gaseous hydrochloric acid combines with lactose, forming a grey mass from which sulphuric acid displaces the sugar. Boiled with nitric acid, lactose is converted into mucic, oxalic, saccharic, and tartaric acids.

Under the action of ferments, lactose undergoes the alcoholic fermentation, but is more susceptible to the lactic. Treated by sodium amalgam, lactose fixes hydrogen, and is transformed into a mixture of equal weights of two hexatomic isomeric alcohols  $C_6H_8(OH)_6$ , *mannite* and *dulcite*—



## SUGAR ANALYSIS.

*Polariscope.*—There are many modifications of this instrument. Some forms of the instrument may be used with *ordinary white light*, whilst others can only be used with *monochromatic light*, e.g., the yellow rays of incandescent sodium. The principles on which they are graduated and adjusted also differ, and are in all cases more or less arbitrary. One class is graduated and adjusted by the displacement of the polarised ray produced by a quartz plate of a certain thickness, whereas in another form the graduation and adjustment is produced by an arbitrary quantity of pure sucrose, dissolved and made up to a definite volume in a certain well-defined length of tube. Uniformity of graduation and adjustment are much to be desired. It is not easy to determine in any given way what method of graduation has been followed in the standardisation of any optical saccharimeter which may be in the analyst's hands. The truly scientific method of graduating an optical saccharimeter would be the use of the absolute cubic centimetre, i.e., the volume occupied by 1 gramme of water weighed *in vacuo* at 4° C. and the use of absolutely pure sugar weighed *in vacuo* for fixing the 100° point on the scale. *Weighing in air and in vacuo.*—The variations which may be caused by weighing in air instead of weighing *in vacuo* must also be taken into account. A flask standardised by weighing the calculated amount of water into it in air will be to a true 100 c.c. flask graduated *in vacuo* as 100.155 c.c. is to 100 c.c. The error of weighing the sugar in air may also be deduced from the following proportion, namely, 26.048 grammes of sugar weighed out in air are equivalent to 26.064 grammes weighed *in vacuo*. In making up a sugar solution weighing 26.048 grammes in the air, and standardising the flask in the air, each c.c. of the solution will contain 0.260236 gramme of sugar. A Mohr flask, graduated to hold 100 grammes of water at 17.5° C., has the following relation to a true 100 c.c. flask, namely, 100.12 : 100 c.c. Instead of a true 100 c.c. flask at this temperature holding 100 grammes as a Mohr flask does, it holds exactly 99.8747 grammes of water. A certificate should be obtained from the maker stating in the case of each instrument the method of its graduation, and then use with that instrument the same flask and same weights which were employed in obtaining this graduation. If the instrument be used with a Mohr flask containing 100

grammes of water weighed in air at a temperature of  $4^{\circ}$ , then all flasks should be graduated in this way, whether they be weighed in air or *vacuo*. In the same way, if the standard weights of sugar used in graduating the instrument are weighed in air, we should make our weighings in air; if, however, the standard weights of sugar are corrected for weight in *vacuo*, then we should also correct for weight in *vacuo*.

TABLE LXIII.—GRADUATION AND ADJUSTMENT OF POLARISCOPES: FRENCH AND GERMAN SCALES.

Substance.	Specific Rotary Power.	French (A).		French (B).		German.	
		Official Formula.		New corrected Formula.		Ventzke's Scale.	
		Metric Graduation.	Mohr's Graduation.	Metric Graduation.	Mohr's Graduation.	Metric Graduation.	Mohr's Graduation.
	+ 0	grms.	grms.	grms.	grms.	grms.	grms.
Cane sugar . . .	66.51	16.19	16.23	16.29	16.33	26.000	26.048
Glucose (dextrose) . .	52.74	20.40	20.45	20.49	20.54	32.765	32.820
Lactose (milk sugar)	52.53	20.51	20.56	20.60	20.65	32.970	33.025
Maltose . . .	138.80	7.78	7.80	7.82	7.84	12.510	12.560
Soluble Starch and dextrine . . .	194.80	5.50	5.51	5.51	5.53	8.885	8.860

1 French degree (Laurent's type) =  $0.6277$  Ventzke =  $0.2167$  arc degrees.

1 German degree (Ventzke's type) =  $1.5931$  Laurent =  $0.3440$  arc degrees.

To describe all the various forms of polariscopes would only confuse the reader; we shall therefore confine ourselves to one single type of instrument, namely, the half-shadow instrument made by Franz Schmidt & Haensch, of Berlin (Figs. 106, 107), which may be used with white light from either paraffin oil or gas lamps. It is handy to manipulate, and its indications are easily read, even by one who is colour-blind. This form of instrument is adjusted to the Ventzke scale, in which  $1^{\circ}$  = the  $\frac{1}{100}$  part of the rotation produced in the plane of polarisation of white light in a column 200 millimetres long by a standard solution of chemically pure sucrose at  $17^{\circ}.5$  C., the standard solution of sucrose in distilled water being such as to contain at  $17^{\circ}.5$  C.  $26.048$  grammes of sucrose in 100 c.c., the weights and volumes being considered as absolute, all weighings being referred to a vacuum. The above definition should be supplemented with a statement of the equivalent circular rotation in degrees, minutes and seconds that would be produced by the standard solution of sugar used to read  $100^{\circ}$  on the scale. The in-

struments should be adjusted by means of control quartz plates, three different plates being used for complete adjustment—one reading approximately  $100^\circ$  on the scale, one  $90^\circ$  and one  $80^\circ$ . These control plates should have their exact value ascertained, in the terms of the Ventzke scale, by comparison with the standard quartz plates, in strict accordance with the foregoing definition, and should also be accompanied by tables giving their value from  $10^\circ$  to  $85^\circ$  C. The weights used should be of solid brass, and should be standardised. The flasks should be of such a capacity as to contain at  $17.5^\circ$  C. 100.06 c.c. when filled, in such a manner that the lowest point of the meniscus of the surface of the liquid just touches the graduation mark. The flasks are standardised to contain this volume, in order that the results shall conform to the scale recommended for adoption without numerical reduction of the weighings to *vacuo*. They should be standardised. *Tubes*.—These should be of brass or glass, 200 and 100 millimetres in length and should also be standardised. *Balances* should be sensitive to 1 milligramme.

*Arrangement of laboratories for polarisation work*.—This has an important bearing on the accuracy of the results. The reading is more readily and accurately taken if the eye of the observer is screened from diffused daylight (as in Fig. 4), therefore a partial darkening of the room, which may be accomplished by means of curtains or hangings, is an advantage. But the temperature at which the observation is made also affects the results, so that the arrangements for darkening the room must not interfere with its proper ventilation; otherwise the heat from the lamps used, if confined within a small room, will cause considerable variations in the temperature of the room from time to time. The proper conditions will possibly best be met by placing the lamps either in a separate room from that in which the instruments are and perforating the wall or partition between the two rooms for the light to reach the end of the instruments, or in a ventilated hood with the walls perforated in a like manner. By lining the wall or partition on both sides with asbestos paper, and inserting a plate of plane glass in the aperture through which the light passes, the increase of temperature from the radiation of the lamp will be still further avoided. With the lamps separated from the instruments in this manner, the space in which the instruments are contained is readily darkened without much danger of its temperature being unduly raised. Some light, of course, is necessary for reading the scales, and if artificial light is employed the sources chosen should be such that as little heat as possible will be generated by them. Small incandescent electric lights are best. Refinements of this kind cannot always be used, but the prime requisite with reference to the avoidance of temperature errors is that all operations—filling the flasks and tubes, reading the solutions, controlling the instrument with standard quartz



plates, etc.—should be done at one and the same temperature, and that this temperature be a constant one, that is, not varying greatly at different hours of the day; *e.g.*, the room should not be allowed to become cold at night, so that it is at low temperature in the morning when work is begun, and then rapidly heated up during the day. The polariscope should not be exposed to the direct rays of the sun during part of the day, and should not be near artificial sources of heat, such as steam boilers, furnaces, flues, etc. The tables upon which the instruments stand should be level.

*Methods of manipulation.*—The methods of manipulation used in the polarisation of sugar are important. They consist in weighing out the sugar, dissolving it, clarifying the solution, making it up to standard volume, filtering, filling the observation tube, regulating the illumination and making the polariscopic reading. The proper conduct of these processes, with the use of accurately graduated

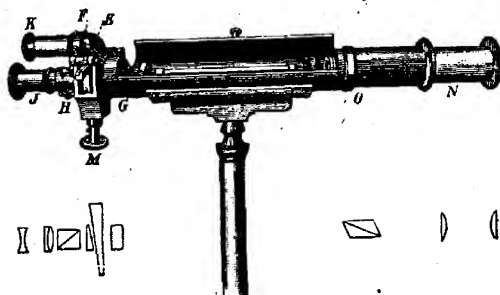


FIG. 106.—Haensch's half-shadow saccharimeter.

apparatus, is the only surety against numerous sources of error. Different sugars require different treatment in classification, and much must necessarily be left to the judgment and experience of the operator. The following directions are based upon various official procedures, such as the one used in the United States custom houses, the method prescribed by the German Government, etc. They embody also the results of recent research in regard to sources of error in the polarimetric estimation of sugar. The instrument employed is known as the half-shadow apparatus of Schmidt & Haensch. It is shown in Fig. 106. The tube N contains the illuminating system of lenses, and is placed next to the lamp; the polarising prism is at O and the analysing prism at H. The quartz wedge compensating system is contained in the portions of the tube marked F, E, G, and is controlled by the milled head M. The tube J carries a small telescope, through which the field of the in-

strument is viewed, and just above is the reading tube K, provided with a mirror and magnifying lens for reading the scale. The tube containing the sugar solution is shown in position in the trough between the two ends of the instrument. In using the instrument, the lamp is placed at a distance of at least 200 millimetres from the end; the observer seats himself at the opposite end so as to bring his eye in line with the tube J. The telescope is moved in or out until the proper focus is secured, so as to give a clearly defined image, when the field of the instrument will appear as a round, luminous disc, divided into two halves by a vertical line passing through the centre, and darker on one half of the disc than on the other. If the observer, still looking through the telescope, will now grasp the milled head M and rotate it, first one way and then the other, he will find that the appearance of the field changes, and at a certain point the dark half becomes light and the light half dark. By rotating the milled head delicately backwards and forwards over this point he will be able to find the exact position of the quartz operated by it, in which the field is natural or of the same intensity of light on both halves. The three different appearances presented by the field are best shown in the following diagram:—

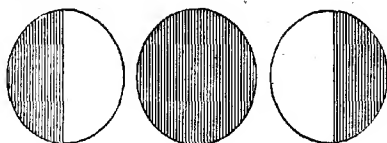


FIG. 107.

With the milled head set at the point which gives the appearance of the middle disc as shown above, the eye of the observer is raised to the reading tube K, and the position of the scale is noted. The scale proper is attached to the quartz wedge, which is moved by the milled head, and attached to the other quartz is a small scale called a vernier, which is fixed, serving for the exact determination of the movable scale with reference to it. On each side of the zero line of the vernier a space corresponding to nine divisions of the movable scale is divided into ten equal parts. By this device the fractional part of a degree indicated by the position of the zero line is ascertained in tenths; it is only necessary to count from zero until a line is found which makes a continuous line with one on the movable scale. With the neutral field, as indicated above, the zero of the movable scale should correspond closely with the zero of the vernier, unless the zero point is out of adjustment. If the observer desires to secure an exact adjustment of the zero of the scale, or in any case if the latter deviates more than one-half of

a degree, the zero lines are made to coincide by moving the milled head and securing a neutral field at this point by means of the small key which comes with the instrument, and which fits a small nipple on the left side of F, the fixed quartz wedge of the compensating system. This nipple must not be confounded with a similar nipple on the right-hand side of the analysing prism H, which it fits as well, but which must never be touched, as the adjustment of the instrument would be seriously disturbed by moving it. With the key on the proper nipple it is turned one way or the other until the field is neutral. Unless the deviation of the zero be greater than  $0^{\circ}5$  it will not be necessary to use the key, but only to note the amount of the deviation, and for this purpose the observer must not be content with a single setting, but must perform the operation five or six times and take the mean of these different readings. If one or more of the readings show a deviation of more than  $0^{\circ}3$  from the general average they should be regarded as incorrect. Between each reading the eye should be allowed ten to twenty seconds of rest. The "setting" of the zero having been performed as above, the determination of the accurate adjustment of the instrument by means of the control quartz plates is proceeded with. Three such plates will be furnished with each polariscope which have sugar values respectively approximating  $80^{\circ}$ ,  $90^{\circ}$  and  $100^{\circ}$ . These values vary with the temperature, and tables are furnished with them giving their exact value at different temperatures from  $10^{\circ}$  to  $35^{\circ}$  C. One of these plates is placed in the instrument and the field observed. The milled head is turned to the right until the exact point of neutrality is re-established, just described above in setting the zero. The scale is read, the observation repeated, the reading again taken, and so on until five or six readings have been made. The average is taken, readings being rejected which show a divergence of more than  $0^{\circ}3$ , and the result corrected for the deviation of the zero point, if any was found, the deviation being added if it was to the left, and subtracted if to the right. If the adjustment of the instrument be correct, the result should be the value of the control plate used as ascertained from the table for the temperature of  $20^{\circ}$  C. Each of the three plates is read in the instrument in this way. A variation of  $0^{\circ}3$  from the established values may be allowed for errors of observation, but in the hands of a careful observer a deviation greater than this with one of the three plates, after a careful setting of the zero, shows that the instrument is not accurately adjusted. The complete verification of the accurate adjustment of the polariscope by means of three control plates as given above should be employed wherever it is set up for the first time by those about to use it, whenever it has sustained any serious shock or injury, and whenever it has been transported from one place to another. It should also be done at least once a week

when the instrument is in constant use. After the complete verification has been performed as described, further checking of the instrument is done by means of one control plate alone, the one approximating  $90^\circ$ , and the setting of the zero point is dispensed with, the indication of the scale for sugar solutions being corrected by the amount of deviation shown in the reading of the  $90^\circ$  control plate from its established value as ascertained from the table at the temperature of the room. For example, a sugar solution polarises  $80^\circ.5$ ; the control plate just before had given a polarisation of  $91^\circ.4$ , the temperature of the room during both observations being  $25^\circ\text{C}$ . According to the table, the value of the control plate at  $25^\circ\text{C}$ . is  $91^\circ.7$ ; the reading is therefore  $0^\circ.3$  too low, and  $0^\circ.3$  is added to the reading of the sugar solution, making the corrected  $80^\circ.8$ . The temperature of the room should be ascertained from a standardised thermometer placed close to the instrument, and in such a position as to be subject to the same conditions. For the daily control of an optical saccharimeter there is perhaps nothing superior,

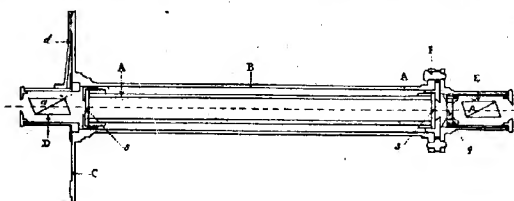


FIG. 103.—Zeiss's portable saccharimeter.

says Wiley, to a first-class quartz plate, the value of which has been tested against pure sugar. The danger of using a quartz plate at its marked value is very great indeed. The quartz plate also has different values according to the position in which it is placed in the instrument, so that its value should be fixed once for all by comparison against a pure sugar solution. A control tube may also be used, which can be made of variable length by ratchet and scale over a carefully graduated vernier, using pure sugar and solution for charging the tubes. An optical saccharimeter should thus be carefully graduated at each  $5^\circ$  from zero to  $100^\circ$ , and any inaccuracy in the scale noted and allowed for in actual practice.

*Portable saccharimeter.*—The instrument by Carl Zeiss of Jena (Fig. 108) consists of a glass tube A of exactly 200 millimetres length, closed by parallel plates of glass *s, s*, at both ends, which is inserted into a brass tube B. This brass tube carries a divided circle c, divided into half-degrees, and upon this circle a shorter tube n rotates and contains a Nicol prism *a* (the analyser). The rotation of n is measured on the divided circle by means of an index pointer

*d.* The other end of *B* bears, by means of an eccentric pivot, a short tube *E* with another Nicol prism *p* (the polariser), and a double plate of rock crystal *q*.

If *E* be turned aside round the pivot the tube *B* is opened and the glass tube *A* can be slipped out, whilst *A* is held in its place when *E* is in its concentric position.

To examine the rotatory power of a solution, open the tube *B* by turning aside *E* and slip out the glass tube *A*; unscrew one of the plates *s* (the tube and both plates *s* having been previously cleaned), and fill the tube in an upright position with the liquid to be examined; then shut it by screwing on the glass plate, taking care that no air-bubbles remain within. Insert the glass tube into *B* and fix it by turning back *E*. Now direct the instrument to the bright sky or other source of light, while looking through the opening at the end of *D*. The clear surface of the rock crystal plate *q* will be seen as a circle with two halves of distinct colours; turn the rotating eye-piece *D* until both semicircles acquire the same colour as near as possible, namely, a reddish violet. This obtained, read off the position of the index pointer *d* on the divided circle. Provided the instrument be properly adjusted, the reading indicates the rotatory power of the solution for a layer of 200 millimetres in degrees and fractions of a degree. In order to test the instrument fill the glass tube with distilled water, turn the index *d* to the zero point of the circle. If the adjustment be correct, the two halves of the plate *q* will appear of the same colour. If it is not adjusted the tube *E* must be turned round its axis within the external tube in which it is inserted, which can be done after loosening a small screw by which it is fixed. In a tube 200 millimetres long a solution of 1 gramme of cane sugar in 100 c.c. of water rotates  $1^{\circ}33$ . Under the same circumstances invert sugar rotates  $-1^{\circ}06$ ;  $1^{\circ}$  indicates, therefore, in the case of cane sugar,  $0.75 (= \frac{3}{4})$  per cent.; and in the case of invert sugar,  $0.94$  per cent., and any number of degrees read off on the divided circle proportionately more. Regarding other substances which may be tested by the instrument, the necessary data must be obtained by experiments or by reference to some work on the subject.

TABLE LXIV.—CORRECTION TO  $17^{\circ}5$  C. OF POLARIMETRIC RESULTS FOR DIFFERENT TEMPERATURES INSTEAD OF 100 POL. DEGS.

° C.	Read.	° C.	Read.	° C.	Read.
15	100.05	18	99.99	22	99.89
16	100.03	19	99.96	23	99.87
17	100.01	20	99.94	24	99.85
17.5	100.00	21	99.90	25	99.82

*Influence of temperature on the specific rotary power of cane sugar.*—The specific rotary power of cane sugar is generally acknowledged to be independent of temperature. If, however, a solution of sugar be made up to the standard volume of 100° C. at a certain temperature, and then polarised at *different* temperatures, it will show either an apparent increase or decrease in rotation power as the temperature is lowered or raised (Table LXIV.). This is simply due to the fact of the contraction of the solvent, thus admitting a larger number of sugar molecules in a given length of the column at a low than at a high temperature.

Waetzel found that the polarisation expressed in degrees of cane sugar in a normal solution (26.048 grammes in 100 c.c.) changes about 0.1 of a degree for each difference of 2° C. in temperature. A solution polarised at 14° C. gave a reading of 95° 6'. The same solution at 34° C. gave a polarisation of 94° 7', or 0.9 for a difference of 20° C. In another experiment a difference of 0.1 of a polarisation degree was shown for a difference of 24° C. On changing the temperature back to the original the original polarisation was obtained. A solution polarised at 17° C. gave 95° 1', but cooled to 10° C. it showed 95° 5'; when reheated to 17° C. it again showed 95° 1'. The above results show the importance of polarising the solution at the temperature at which it is made. For this reason the solutions should all be made at a given temperature in a place near that where the polarisations are made, and the temperature should not be allowed to change from the time the solutions are made until the polarisations are complete.

*Graduation of sugar flasks.*—Wiley points out that most sugar flasks are graduated either at 15° or 17° 5', and that the mean temperature in the U.S.A. is much higher than this, and that it will probably be well to have all U.S.A. flasks graduated at about 22°. Since, however, the volume of a flask will change very little within the limits given, it is more important that the polarisations be made at the same temperature at which the solution is made up, than that all the flasks should be graduated at a given temperature.

TABLE LXV.—EFFECT OF CONCENTRATION ON ROTARY POWER OF SUCROSE (TOLLENS, SCHMITZ AND LANDOLT).

C.	[α] D.	C.	[α] D.	C.	[α] D.	C.	[α] D.
4	66.63	10	66.57	16	66.52	22	66.46
5	66.62	11	66.57	17	66.51	23	66.45
6	66.61	12	66.56	18	66.50	24	66.44
7	66.60	13	66.55	19	66.49	25	66.43
8	66.59	14	66.54	20	66.48	26	66.42
9	66.58	15	66.53	21	66.47	27	66.41
						28	66.40

TABLE LXVI.—ROTARY POWER OF SUGARS.

Substance.	° C.	Limits of Concentration.	Rotary Power $[\alpha]_D$ .	Observers.
Cane sugar, $C_{12}H_{22}O_{11}$ the solution at 17°·5 compared with water at 4°.	20	$p = 4 - 18$	+ 68·810 - 0·01553 <i>p</i> - 0·000052462 <i>p</i> <sup>2</sup>	Tollens.
"	20	$p = 18 - 69$	+ 66·986 + 0·015035 <i>p</i> - 0·00039893 <i>p</i> <sup>2</sup>	"
"	20	$p = 5 - 18$	+ 66·727 - 0·01534 <i>p</i> + 0·00032396 <i>p</i> <sup>2</sup>	"
"	20	$p = 18 - 69$	+ 66·908 + 0·015016 <i>p</i> - 0·00089817 <i>p</i> <sup>2</sup>	Schmitz.
"	20	$q = 85 - 98$	+ 64·156 + 0·05196 <i>q</i> - 0·000280592 <i>q</i> <sup>2</sup>	Landolt.
"	20	$c = 4 - 28$	+ 66·67 - 0·00955 <i>c</i>	Nasini and Villavechia.
"	20	$p = 0·5 - 1·2$	+ 66·96 - 4·6896 <i>p</i> + 1·9815 <i>p</i> <sup>2</sup>	Pribram.
"	20	$p = 3 - 65$	+ 66·438 + 0·010312 <i>p</i> - 0·00085449 <i>p</i> <sup>2</sup>	Hesse.
"	20	$p = 0·2 - 4$	+ 64·262 - 0·6083 <i>p</i> + 2·3402 <i>p</i> <sup>2</sup>	"
"	15	$c = 0 - 10$	+ 68·65 - 0·828 <i>c</i> + 0·115415 <i>c</i> <sup>2</sup> $[\alpha]_D = 75·08$ for $[\alpha]_D = 66·5$	Muntz.
"	15	$c = 5$	+ 67	"
"	15	$c = 10$	+ 67·7 alcoholic solution	"
"	15	$c = 5$	+ 64·9	"
"	15	$c = 10$	+ 61·3	"
"	15	$c = 5$	+ 46·9	"
"	15	$c = 10$	+ 51·8	"
"	15	$c = 5$	+ 66	"
"	15	$c = 5$	+ 0·170	"
"	15	$c = 5$	+ 0·140	"
"	15	$c = 5$	+ 52·50 + 0·018796 <i>p</i> + 0·00061683 <i>p</i> <sup>2</sup>	"
"	15	$p = 0 - 100$	+ 47·73 + 0·015634 <i>p</i> + 0·00039889 <i>p</i> <sup>2</sup>	"
"	15	$p = 0 - 100$	+ 61·78	"
"	15	$c = 8$	+ 61·67	"
"	15	$c = 3$	+ 61·67	"
"	15	$c = 2·5$	+ 51·80	"
"	15	$c = 2·5$	+ 49·25	"
"	15	$c = 2·0$	+ 49·25	"
Cane sugar, $C_{12}H_{22}O_{11}$ the solution at 17°·5 compared with water at 17°·5	20	$p = 4 - 18$	+ 68·810 - 0·01553 <i>p</i> - 0·000052462 <i>p</i> <sup>2</sup>	Tollens.
"	20	$p = 18 - 69$	+ 66·986 + 0·015035 <i>p</i> - 0·00039893 <i>p</i> <sup>2</sup>	"
"	20	$p = 5 - 18$	+ 66·727 - 0·01534 <i>p</i> + 0·00032396 <i>p</i> <sup>2</sup>	"
"	20	$p = 18 - 69$	+ 66·908 + 0·015016 <i>p</i> - 0·00089817 <i>p</i> <sup>2</sup>	Schmitz.
"	20	$q = 85 - 98$	+ 64·156 + 0·05196 <i>q</i> - 0·000280592 <i>q</i> <sup>2</sup>	Landolt.
"	20	$c = 4 - 28$	+ 66·67 - 0·00955 <i>c</i>	Nasini and Villavechia.
"	20	$p = 0·5 - 1·2$	+ 66·96 - 4·6896 <i>p</i> + 1·9815 <i>p</i> <sup>2</sup>	Pribram.
"	20	$p = 3 - 65$	+ 66·438 + 0·010312 <i>p</i> - 0·00085449 <i>p</i> <sup>2</sup>	Hesse.
"	20	$p = 0·2 - 4$	+ 64·262 - 0·6083 <i>p</i> + 2·3402 <i>p</i> <sup>2</sup>	"
"	15	$c = 0 - 10$	+ 68·65 - 0·828 <i>c</i> + 0·115415 <i>c</i> <sup>2</sup> $[\alpha]_D = 75·08$ for $[\alpha]_D = 66·5$	Muntz.
"	15	$c = 5$	+ 67	"
"	15	$c = 10$	+ 67·7 alcoholic solution	"
"	15	$c = 5$	+ 64·9	"
"	15	$c = 10$	+ 61·3	"
"	15	$c = 5$	+ 46·9	"
"	15	$c = 10$	+ 51·8	"
"	15	$c = 5$	+ 66	"
"	15	$c = 5$	+ 0·170	"
"	15	$c = 5$	+ 0·140	"
"	15	$c = 5$	+ 52·50 + 0·018796 <i>p</i> + 0·00061683 <i>p</i> <sup>2</sup>	"
"	15	$p = 0 - 100$	+ 47·73 + 0·015634 <i>p</i> + 0·00039889 <i>p</i> <sup>2</sup>	"
"	15	$p = 0 - 100$	+ 61·78	"
"	15	$c = 8$	+ 61·67	"
"	15	$c = 3$	+ 61·67	"
"	15	$c = 2·5$	+ 51·80	"
"	15	$c = 2·5$	+ 49·25	"
"	15	$c = 2·0$	+ 49·25	"
Dextrose anhydrous, $C_6H_{12}O_5$ $C_6H_{12}O_5 \cdot H_2O$	20	$p = 0 - 100$	+ 47·73 + 0·015634 <i>p</i> + 0·00039889 <i>p</i> <sup>2</sup>	"
"	15	$p = 0 - 100$	+ 61·78	"
"	15	$c = 8$	+ 61·67	"
"	15	$c = 3$	+ 61·67	"
"	15	$c = 2·5$	+ 51·80	"
"	15	$c = 2·5$	+ 49·25	"
"	15	$c = 2·0$	+ 49·25	"
"	15	$c = 2·0$	+ 49·25	"
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"	15	$c = 2·0$	+ 49·25	"
"	15	$c = 2·0$	+ 49·25	"
"	15	$c = 2·0$	+ 49·2	

Glucose, $C_6H_{12}O_6 + H_2O$	20	$p = 8 - 21$	$+ 47.925 + 0.01534p + 0.000388p^2$	Tollens.
"	20	$p = 7 - 83$	$+ 52.718 + 0.01708p + 0.0004271p^2$	"
"	15	$c = 6.9$	$[a]_D^{20} = -106$	Jodin.
	14		$[a]_D^{20} = -79.5$	Dubrunfaut.
	52		$[a]_D^{20} = -53$	"
	90		$[a]_D^{20} = -100$	Neubauer.
	14		$[a]_D^{20} = -104$	Jodin.
Levulose, $C_6H_{12}O_6$	0 - 40	$c = 0 - 40$	$- 100.8 - 0.105c + 0.56c^2$	Jungfleisch and Grimbert.
	20	$c = 1 - 14$	$+ 20.07 - 0.041c$	Hammer Schmid.
	14		$[a]_D^{20} = -26.65$	Dubrunfaut.
	52		$[a]_D^{20} = -13.92$	"
	30		$[a]_D^{20} = 0$	Tuchsmid.
Invert sugar, $C_{12}H_{22}O_{11}$	0	$c = 17 - 21$	$- 27.30/D = - (27.9 - 0.82d)$	Berthelot.
Lactose, $C_{12}H_{22}O_{11} + H_2O$	20	$p = 0 - 86$	$+ 52.47 \text{ constant}$	Hesse.
"	15	$c = 2 - 12$	$+ 54.54 - 0.547p + 0.06475c^2$	Ost.
Maltose, $C_{12}H_{22}O_{11}$	20	$p = 0 - 96$	$+ 137.04$	Meist.
Galactose, $C_{12}H_{22}O_{11}$	10 - 30	$p = 5 - 85$	$+ 83.883 + 0.0785p - 0.208d$	Von Lippmann,
Arabinose, $C_{12}H_{22}O_{11}$	20	$p = 10$	$+ 105.5 \text{ constant}$	etc.
Arabinic acid, $C_{12}H_{20}O_{11}$		$c = 5$	$[a]_D^{20} = + 37.9 + 46.1 - 28.8 - 29.2 - 30$	Scheibler.
Raffinose, $C_{18}H_{34}O_{16} + 5H_2O$	20	$p = 0 - 10$	$+ 104.5 \text{ constant}$	Loiseau, etc.
Tetraacetylglucose, $C_6H_7(C_2H_3O)_4O_{11}$		$c = 7.46$	$[a]_D^{20} = + 173.2$	Schäfer, Berger.
Myrcose, $C_6H_7O_{11} + 2H_2O$		$c = 10.03$	$[a]_D^{20} = + 199$	Mitscherlich.
Trehalose, $C_{12}H_{22}O_{11} + 2H_2O$	15	$c = 8.4 \text{ to } 14.8$	$[a]_D^{20} = + 88$	Berthelot.
Maltitose, $C_6H_7O_{11} + 3H_2O$	25	$c = 17.27$	$[a]_D^{20} = + 94.1 \text{ (anhydrous)}$	"
Malezitose, $C_6H_7O_{11} + H_2O$	20	$c = 18.6$	$[a]_D^{20} = -46.9$	"
Sorbit, $C_6H_{12}O_6$		$c = 23.9$	$[a]_D^{20} = -0.03$	Pasteur.
Mannit, $C_6H_{12}O_6$		$c = 15$	$+ 40 \text{ alcoholic solution}$	Krausmann.
Nitromannit, $C_6H_7(O-NO_2)_6$		$c = 7.5$	$[a]_D^{20} = 47.6$	Hirschweiz.
Leululose, $C_6H_{12}O_6 + H_2O$		$c = 10.2$		Pruiner.
Quercit, $C_6H_{12}O_6$	16	$c = 1 \text{ to } 10$	$+ 24.3$	



*Volumetric analysis of sugars.*—It has been claimed for this method that it is the most expeditious, the easiest, and when well performed the most exact process of sugar analysis. The principle of the method depends on the three following facts: 1. Sucrose has no action on the special copper solution known as Bareswill's liquor (Fehling's solution). 2. All *glucoses*, on the contrary, reduce this solution and precipitate its copper as cuprous oxide. 3. Sucrose which has no action on the boiling solution of copper, fixes water under the action of heat and mineral acids (hydrochloric and sulphuric), and is transformed in equal proportion into two new sugars, glucose and levulose, a mixture which has been termed *invert sugar* (p. 338).

*Reagents.*—1. *Sulphuric acid for inversion.*—100 grammes of sulphuric acid are run in a thin stream into 200 grammes of distilled water with constant stirring. The mixture becomes very hot. When it is cold it is preserved in a well-stoppered bottle. 2. *Normal sugar solution  $\frac{1}{2}$  per cent.*—1 gramme of pure dry sugar candy is dissolved in 50 c.c. of distilled water, to which about 2 c.c. of sulphuric acid are added to invert the sugar by boiling for a quarter of an hour. The operation is performed in a graduated flask of 200 c.c. capacity. When the solution is cold it is made up to 200 c.c., and closed with the thumb, and the flask turned repeatedly upside down so as to thoroughly mix the solution and render it uniform throughout. This solution therefore contains  $\frac{1}{2}$  per cent. of cane sugar in the form of invert sugar. 3. *Copper solution for the estimation proper.*—Particular care must be devoted to the preparation of this liquid. *Solution 1.*—36.46 grammes of sulphate of copper are dissolved in 140 c.c. of distilled water. *Solution 2.*—200 grammes of the neutral tartrate of potash and soda are dissolved in 600 c.c. of caustic soda of 22° B., sp. gr. 1.20. The second solution is run into a 1 litre flask, and the first solution added in small portions at a time; finally, the bulk is made up to a litre containing the sulphate of copper and the double tartrate of potash and soda. If the sulphate of copper was pure enough 10 c.c. would require for its reduction 0.050 gramme of cane sugar, which would correspond to 0.0526 gramme of glucose. But it is necessary to verify it. By a 10 c.c. pipette 10 c.c. of the copper solution are run into a small Bohemian glass flask of 100 c.c. capacity, or, better still, into a test-tube containing a few fragments of pumice to prevent bumping on boiling the liquid. The Mohr's burette is filled to the upper division with the standard solution of sugar ( $\frac{1}{2}$  per cent.). This done, the copper solution is rapidly boiled, and the sugar solution run in drop by drop. After each addition the liquid is boiled, whilst the flask is shaken. The liquid at first loses its transparency, becomes yellow and then red, and finally becomes completely decolorised. As this moment draws nigh after each addition of the standard sugar solu-

tion, the flask is placed for an instant on white paper so as to seize the precise moment when the liquid has become perfectly colourless. A final drop of sugar solution is then added to make sure that no precipitate is formed, and when that result is obtained the operation is finished. The volume of liquid is then read off from the scale on the burette. Suppose that the number of divisions were equal to 10 c.c., then—

$$\begin{array}{cccc} \text{C.c.} & \text{C.c.} & \text{Gramme.} & \text{Gramme.} \\ 100 : 10 :: 0.50 : x = 0.05 \end{array}$$

The strength of the standard cupro-potassic solution is therefore exact. The formula adopted is that of Violette, which is to be preferred to Fehling's or Mohr's, as the solution keeps better. The real titrations are simply repetitions of the first experiment.

*Volumetric estimation of sugar.*—The complete analysis of a sugar ought to give—1. The percentage of glucoses. 2. The percentage of cane sugar. 3. Moisture and impurities. These results are obtained by two consecutive experiments. *Total sugar.*—1 gramme of the sugar is dissolved in about 50 c.c. of distilled water, to which 2 to 3 c.c. of acid are added for inversion. The solution is boiled for a quarter of an hour over a spirit lamp or Bunsen burner. The heat is then withdrawn and the solution allowed to cool, and then filtered into a 200 c.c. flask. The first flask in which the solution was dissolved is very carefully washed, and the wash water run through the filter so as to remove all trace of sugar. The filtrate is then made up to 200 c.c. with distilled water. The mouth of the flask is then closed with the thumb of the right hand, and turned repeatedly upside down so as to obtain a perfectly homogeneous solution. The Mohr's burette having been carefully washed with distilled water and dried by a tube enveloped in filter paper, made to glide along its inside surface, is filled with the standard sugar solution, and a new test is made similar to the first. Suppose 10.8 c.c. of the sugar solution were required to reduce 10 c.c. of the cupro-potassic solution, we then get—

$$\begin{array}{cccc} \text{C.c.} & \text{C.c.} & \text{Gramme.} & \text{Gramme.} \\ 10.8 : 200 :: 0.050 : x = 0.9259 \end{array}$$

1 gramme of the sample therefore contained 0.9259 gramme of sugar, say 92.59 per cent. Table LXVII. obviates calculation. It now remains to be seen whether the sample contained any glucose. That point is determined as follows. \* *Estimation of glucose.*—A fresh amount of sugar—10 grammes—is again weighed out and dissolved in about 50 c.c. of water, and filtered as before into the graduated flask, and brought by the addition of the wash water to 100 c.c. only. The flask is turned upside down repeatedly to mix the whole of the layers of liquid. The Mohr's burette having been washed and dried is filled with the sugar solution, and a fresh titration with

the cupro-potassic solution is performed. Suppose that 16.1 c.c. of sugar solution were required to reduce 10 c.c. of cupro-potassic solution, we get—

$$\begin{array}{rcl} \text{C.c.} & \text{C.c.} & \text{Gramme.} \\ 16.1 : 100 :: 0.050 : x = 0.310 \end{array}$$

Now 0.310 gramme for 10 grammes of sugar = 3.10 per cent. of glucose expressed as cane sugar.

		Result.
Total sugar	.	92.69 per cent.
Glucose	.	8.10 "
Difference	.	89.49 "

The difference gives the amount of cane sugar.

To arrive definitely at the exact composition of the sugar analysed, the result of the second experiment, in which the glucose is expressed as cane sugar, is calculated into actual glucose,  $C_6H_{12}O_6$ . This is done by multiplying the result by 1.052—

$$3.10 \times 1.052 = 3.26.$$

#### COMPOSITION OF THE SUGAR ANALYSED.

Cane sugar	.	89.49 per cent.
Glucose <sup>1</sup>	.	3.26 "
		92.75 "
Moisture and impurities by difference	.	7.25 "
		100.00 "

Table LXVII. avoids the necessity of calculations.

TABLE LXVIa.—ANALYSES OF COMMERCIAL STARCH GLUCOSE (LINDET).

	Solid.	Crystal Syrup.
	Per Cent.	Per Cent.
Water . . .	20.44	20.63
Glucose . . .	73.42	35.35
Dextrine . . .	5.78	43.83
Ash . . .	0.35	0.19
	99.99	100.00

<sup>1</sup> Used in a generic sense throughout the description of this process.

TABLE LXVII.—PROPORTION OF CANE SUGAR CONTAINED IN IMPURE SUGARS OR SACCHARINE JUICES.

I.  $\frac{1000}{V}P$  100. II.  $\frac{50}{V}P$  100. (VILLE.)

I. Rich Sugars.		II. Saccharine Juices.		Recapitulation of Principles.
Volume of Sugar Solution used.	Cane Sugar per Cent.	Volume of Sugar Solution used.	Cane Sugar per Cent.	
c.c.		c.c.		
10	100.0	1	50	I.
10.1	99.01	$1\frac{1}{2}$	83.33	10 molecules of cane sugar produce 20 molecules of glucose. (See equation.)
10.2	98.04	2	25	II.
10.3	97.09	$2\frac{1}{2}$	20	1 part of cane sugar corresponds to 1.052 parts of glucose.
10.4	96.15	3	16.07	III.
10.5	95.24	$3\frac{1}{2}$	14.29	Cane sugar has no action on Fehling's solution.
10.6	94.34	4	12.50	IV.
10.7	93.46	$4\frac{1}{2}$	11.11	Glucoses precipitate Fehling's solution as cuprous oxide.
10.8	92.59	5	10	V.
10.9	91.74	$5\frac{1}{2}$	9.09	One equivalent of glucose ( $C_6H_{12}O_6$ ) precipitates 10 equivalents of sulphate of copper ( $CuSO_4, 5H_2O$ ).
11	90.91	6	8.33	180 grammes of glucose correspond to 1246.8 of sulphate of copper, and 5 grammes of glucose correspond to 34.633 grammes of sulphate of copper.
11.1	90.09	$6\frac{1}{2}$	7.69	VI.
11.2	89.29	7	7.14	10 c.c. of Fehling's solution correspond to 0.05 gramme of cane sugar.
11.3	88.50	$7\frac{1}{2}$	6.67	VII.
11.4	87.72	8	6.25	Mineral acids by the aid of heat convert cane sugar into glucose— $C_{12}H_{22}O_{11} + H_2O = 2C_6H_{12}O_6$
11.5	86.96	$8\frac{1}{2}$	5.88	VIII.
11.6	86.21	9	5.56	Inverted sugar consists of two isomeric sugars, but of different optical properties—
11.7	85.47	$9\frac{1}{2}$	5.26	1. Glucose properly so called, which deviates the plane of polarised light $56^\circ$ to the right.
11.8	84.75	10	5	2. Levulose, which deviates polarised light $106^\circ$ to the left.
11.9	84.03	$10\frac{1}{2}$	4.76	Column I. refers to a solution of 1 gramme of cane sugar in 200 c.c. of distilled water.
12	83.33	11	4.55	
12.1	82.64	12	4.17	
12.2	81.97	13	3.85	
12.3	81.30	14	3.57	
12.4	80.64	15	3.33	
12.5	80	16	3.12	
12.6	79.36	17	2.94	
12.7	78.74	18	2.78	
12.8	78.12	19	2.63	
12.9	77.52	20	2.50	
13	76.92	$22\frac{1}{2}$	2.22	
13.1	76.34	25	2	
13.2	75.76	$27\frac{1}{2}$	1.82	
13.3	75.19	30	1.67	
13.4	74.64	$32\frac{1}{2}$	1.54	
13.5	74.07	35	1.43	
13.6	73.53	$37\frac{1}{2}$	1.33	
13.7	72.99	40	1.25	
13.8	72.46	$42\frac{1}{2}$	1.18	
13.9	71.94	45	1.11	
14	71.43	$47\frac{1}{2}$	1.06	
...	...	50	1	

TABLE LXVIII.—PHENOMENA OBSERVED IN TITRATING GLUCOSE WITH COPPER SOLUTION (VIOLETTE).

Phase of the Operation.	Fundamental Characteristics.		Secondary Characteristics.	
	Precipitation on the Surface of the Hot Clear Liquid.	Coloration on the Surface of the Hot Clear Liquid.	Colour of the Mass in Full Boil.	Colour of the Liquid Floating on the Deposit.
Commencement.	Yellow precipitate, then orange, then red.	Same shades as that of the whole liquid as given in the fifth column.	Deep blue violet.	Deep blue.
Towards the middle.	Orange red precipitate very perceptible.		Reddish violet.	Light blue.
Towards the end.	Orange red precipitate.		Brick red.	Very pale blue.
" "	Slight cloudiness.		Decided red.	"
End.	Final cloudiness scarcely perceptible.		Bright red.	Scarcely perceptible blue.
Beyond the finish.	No precipitate.	Amber transparent zone.	Beautiful bright red.	Colourless.
" "	"	Transparent zone, deeper coloured.	" "	Straw yellow.
" "	"	Transparent zone, colour of gamboge.	" "	Amber.
" "	"	Deep brown transparent zone.	Bright red, a little yellow.	Deeper.
" "	"		Red, slightly brown.	Gamboge yellow.

*Note.*—The froth of the liquid on boiling, at first blue, becomes colourless at the end of the operation, then yellow and brownish yellow when the precipitation is gone beyond. The liquid then gives off a burnt-sugar smell.

*Politis's rapid method for the estimation of sugar with  $\frac{N}{10}$  copper-*

*alkali solution.*—The determination of sugar with Fehling's solution would be accurate and rapid enough if the end of the reaction—that is, the complete decolorisation of the alkaline copper solution—was easy to determine. The suspended copper suboxide makes this very difficult. To avoid this inconvenience and to make the determination more accurate and rapid, the author uses a definite amount of sugar solution, an excess of  $\frac{N}{10}$  copper solution, and estimates the

TABLE LXIX.—ESTIMATION OF GLUCOSE IN RAW SUGAR.

Solution of 16.29 Grammes Sugar in 100 c.c.				Sugar Solution treated with 10 per Cent. of Carbonate of Soda.			
Working on 1 c.c. of Fehling's Solution.		Working on $\frac{1}{2}$ c.c. of Fehling's Solution.		Working on 1 c.c. of Fehling's Solution.		Working on $\frac{1}{2}$ c.c. of Fehling's Solution.	
C.c. Sugar Solution.	Glucose per Cent.	C.c. Sugar Solution.	Glucose per Cent.	C.c. Sugar Solution.	Glucose per Cent.	C.c. Sugar Solution.	Glucose per Cent.
5.1	0.60	2.6	0.60	5.7	0.60	2.9	0.60
5.6	0.55	2.8	0.55	6.2	0.55	3.1	0.55
6.2	0.50	3.1	0.50	6.8	0.50	3.4	0.50
6.9	0.45	3.5	0.45	7.5	0.45	3.6	0.45
7.7	0.40	3.9	0.40	8.5	0.40	4.3	0.40
8.8	0.35	4.4	0.35	9.7	0.35	4.9	0.35
10.3	0.30	5.2	0.30	11.3	0.30	5.7	0.30
12.4	0.25	6.2	0.25	13.6	0.25	6.8	0.25
15.5	0.20	7.8	0.20	17.0	0.20	8.5	0.20
20.6	0.15	10.3	0.15	22.7	0.15	11.4	0.15
30.9	0.10	15.5	0.10	34.0	0.10	17.0	0.10
61.8	0.05	30.9	0.05	68.0	0.05	34.0	0.05

excess according to De Haen's method, with potassium iodide and sodium hyposulphite. The following solutions are required :—

(1)  $\frac{N}{10}$  copper solution consisting of 24.95 grammes pure copper sulphate, 140 grammes sodium potassium tartrate, 25 grammes pure sodium hydrate, and sufficient water to make 1 litre.

(2)  $\frac{N}{10}$  solution of sodium hyposulphite, 24.8 grammes per litre.

(3)  $\frac{N}{10}$  iodine solution, 12.7 grammes per litre. The three solutions must correspond exactly, and 1 c.c. of the proper solution is equivalent to 0.0036 gramme glucose. The estimation is made as

follows: 50 c.c. of the copper solution are boiled in a porcelain dish and 10 c.c. of a sugar solution added, which contains about 1 gramme glucose in 1,000. The whole is heated for five minutes, made up to

TABLE LXX.—VOLUMETRIC ESTIMATION OF GLUCOSE AND LEVULOSE, LACTOSE AND MALTOSE.

(Table for use with solutions containing 0.1 to 0.5 per cent. of reducing sugars—titrated with 10 c.c. Fehling's solution—the results being expressed in grammes per litre or in lb. per 100 gallons.)

C.c. Sugar Solution.	Glucose and Levulose.	Lactose (anhydr.).	Maltose (anhydr.).	C.c. Sugar Solution.	Glucose and Levulose.	Lactose (anhydr.).	Maltose (anhydr.).
	Diff.				Diff.		
10	5.000	6.350	7.500	26	1.923	2.442	2.884
	.454				.071		
11	4.546	5.773	6.818	27	1.852	2.354	2.778
	.379				.066		
12	4.167	5.292	6.250	28	1.786	2.268	2.679
	.321				.062		
13	3.846	4.884	5.770	29	1.724	2.189	2.586
	.275				.057		
14	3.571	4.536	5.357	30	1.667	2.117	2.500
	.237				.054		
15	3.334	4.233	5.000	31	1.613	2.049	2.419
	.209				.050		
16	3.125	3.969	4.687	32	1.563	1.984	2.344
	.184				.048		
17	2.941	3.744	4.412	33	1.515	1.924	2.272
	.163				.044		
18	2.778	3.523	4.167	34	1.471	1.868	2.206
	.146				.042		
19	2.632	3.342	3.947	35	1.429	1.814	2.143
	.132				.040		
20	2.500	3.175	3.750	36	1.389	1.764	2.083
	.119				.038		
21	2.381	3.024	3.571	37	1.351	1.717	2.027
	.108				.035		
22	2.273	2.886	3.409	38	1.316	1.671	1.973
	.099				.034		
23	2.174	2.761	3.261	39	1.282	1.628	1.923
	.091				.032		
24	2.083	2.643	3.125	40	1.250	1.588	1.875
	.083						
25	2.000	2.540	3.000				
	.077						

100 c.c., and exactly 50 c.c. filtered into a beaker. The blue liquid is made slightly acid, a slight excess of potassium iodide and some starch solution added, and the iodine estimated by means of the  $\frac{N}{10}$

sodium hyposulphite solution. This iodine corresponds to the un-reduced copper in the liquid, and the copper required by the glucose can be calculated by difference. The number of c.c.  $\frac{N}{10}$   $\text{Na}_2\text{S}_2\text{O}_3$  solution required is deducted from 25, and the result will be the number of c.c. of copper solution which have been reduced by 5 c.c. of the sugar solution. *Example*.—50 c.c.  $\frac{N}{10}$  copper solution were boiled with 10 c.c. of diabetic urine which had been diluted to 5 times its volume, and the estimation completed as above. 11 c.c. of  $\frac{N}{10}$   $\text{Na}_2\text{S}_2\text{O}_3$  were required to completely decolorise the liquid;  $25 - 11 = 14$  c.c. of copper solution which had been reduced by one-half of the sugar solution, what is 5 c.c. of diluted or 1 c.c. of the original urine? As 1 c.c. of copper solution = 0.0036 gramme glucose, 1 c.c. of urine contained 0.0504, or 1 litre 50.4 grammes glucose.

*Soldaini's solution*.—A solution of copper carbonate in potassium carbonate was proposed as a substitute for Fehling's solution by Arturo Soldaini.

*Modifications of Soldaini's method*.—1. *Striegler's modifications*.—Striegler made a number of experiments to find means by which Soldaini's reagent could be prepared with a definite copper content. He first examined Bodenbender and Scheller's method of preparing this reagent, and then elaborated a method by which a constant solution can be prepared. He followed Degener, Schweitzer and Preuss's instructions so as to get accurate results. Scheller's and Preuss's researches are summarised on pp. 374-376. Analyses of various portions of the reagent prepared by the same method show—(1) That almost one-half the potassium carbonate used has been decomposed with the evolution of  $\text{CO}_2$ . (2) That the amount of copper dissolved varies considerably between 131.1 and 189.7 milligrammes copper in 100 c.c.; with regard to Preuss's figures, even from 104 to 187 milligrammes copper in 100 c.c. (3) That the quantity of copper dissolved is very low as compared with the quantity used. Used = 0.727 gramme copper dissolved at the most = 0.1597 gramme copper in 100 c.c.

Further experiments proved that on dissolving the potassium carbonate in hot water a large amount is decomposed, and that in preparing the reagent more is used than can remain in solution at the ordinary temperature. It is therefore necessary, after preparing the reagent, to decompose bicarbonate at least in part, so as to prevent any excess from crystallising out. This can easily be accomplished by boiling. As the result of further experiments, Striegler prepared a reagent of definite composition, as follows: An amount of copper sulphate corresponding to 1.025 gramme copper in 100



c.c. was accurately weighed out, dissolved in cold water, made alkaline with soda lye, and the precipitate washed well. The usual amount of potassium bicarbonate was then added, and the whole heated at  $40^{\circ}$  to  $45^{\circ}$  C. until the bicarbonate had dissolved, and then at  $60^{\circ}$  until complete solution of the hydroxide of copper took place. This operation requires from one and a half to two hours, the preparation of larger quantities occupying a correspondingly larger amount of time. The solution was boiled some time to destroy the bicarbonate, cooled and made up to the original volume. Analyses show, as could be surmised beforehand, that this solution gives constant results; differences which could be ascribed to a varying carbonic acid content did not occur; we must naturally suppose that enough bicarbonate has been destroyed to prevent any crystallisation. The following alternate methods of preparation were finally adopted as serving the purpose; *A*. To prepare 2 litres of the reagent, exactly 12.77 grammes of pure, finely powdered copper sulphate were dissolved in cold water, precipitated with soda lye, and the precipitate collected on a filter. A simple contrivance was used to facilitate washing. A flask holding about three-quarters of a litre was closed with a perforated stopper, through which passed a glass tube about 6 to 7 centimetres long and 5 millimetres wide. The flask was inverted in such a manner over the funnel that the end of the tube was just below the level of the liquid in the filter. This arrangement is automatic and reliable. The washed hydrate was carefully transferred to a porcelain dish, removing the last particles from the filter with the wash bottle, mixed well with a glass rod, and then washed into a 2,500 c.c. flask together with 597.7 grammes potassium bicarbonate. Enough water was added to make the volume about 2,000 c.c. This was heated on the water bath to  $45^{\circ}$  C., frequently shaking until the bicarbonate had dissolved, and then direct heat applied to dissolve the hydrate, not allowing the temperature to rise over  $60^{\circ}$  to  $70^{\circ}$  C. After complete solution the reagent is boiled for one to one and a half hours so as to decompose the bicarbonate, allowed to cool, transferred to a 2-litre flask, made up to the mark, and filtered. It is unnecessary to use a reflux condenser or to make up 2 litres beforehand, as the escape of carbonic acid causes a concentration which must be corrected by the addition of water.

The solution in itself is stable, not even boiling causing decomposition. If, however, it is diluted with 5 times its volume of water a bluish green precipitate appears, which becomes black on boiling. This precipitate is therefore copper oxyhydrate, which is converted into black copper oxide on heating. This observation induced the author to examine the action of water on the usual Soldani reagent. He found that when diluted with about 20 times its volume of water it also gave a precipitate of black oxide upon heating. This seems

to indicate that the copper in Soldaini's reagent is not present as carbonate, but rather as hydrate.

*B.* The behaviour of basic carbonate of copper showed another way of preparing a constant reagent.

The basic carbonate prepared from 50 grammes copper sulphate is heated for five or six hours with 300 grammes potassium carbonate and about 700 c.c. of water, the heat being kept at 45° C. as before until the bicarbonate has dissolved and then at 60° C. The solution is filtered, and the excess of bicarbonate allowed to crystallise. The filtrate contained, in 100 c.c., 0.2882 grammes copper and a quantity of potassium hydrate, corresponding to 36.7 grammes potassium bicarbonate. To prepare 1 litre of Soldaini's reagent (0.1625 gramme Cu and 29.7 grammes  $\text{HKCO}_3$  in 100 c.c.), 564 c.c. of above filtrate are required, which contain 207 grammes of the bicarbonate. It is therefore necessary to add 90 grammes  $\text{HKCO}_3$  or an equivalent amount of  $\text{K}_2\text{CO}_3$ , the use of the latter avoiding subsequent boiling for the purpose of destroying the excess of bicarbonate. A solution thus prepared could not be distinguished from another prepared by the first method. It depends altogether on the conditions whether one or the other should be preferred. If small quantities, for instance 2 litres, are to be prepared, the first is best applied; but if large quantities are required, as in manufacturing laboratories, where a steam bath is always at hand and the time is immaterial, then the last method is best followed.

2. *Ettore Soldaini's modification of Arturo Soldaini's solution.*—To avoid the decomposition of stronger solutions, E. Soldaini proposed the use of a deci-normal solution, made as follows:—3.464 grammes of crystallised copper sulphate ( $\text{CuSO}_4 + 5\text{H}_2\text{O}$ ) and 297 grammes of  $\text{KHCO}_3$  are dissolved in a litre of water. For the estimation of the glucose with the  $\frac{1}{10}$  normal solution 100 c.c. of this solution are heated to boiling, and the glucose solution added gradually until complete reduction takes place. The 100 c.c. of the  $\frac{1}{10}$  normal solution correspond to 0.05 gramme of glucose.

The liability of Soldaini's reagent to decompose when much diluted explained the varying results obtained in the first investigations upon its reduction by invert sugar. Washing with hot water at once induces this decomposition, to avoid which a washing with cold solution of potassium bicarbonate was substituted for the addition of water after boiling, with the invert sugar until all the copper had been removed, following with water to remove all the alkali.

Ettore Soldaini prepares a deci-normal copper solution by dissolving 3.464 grammes  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$  and 297 grammes  $\text{KHCO}_3$  in 1000 c.c. of water. He deems it unnecessary to previously precipitate with  $\text{Na}_2\text{CO}_3$ , and preferably adds the  $\text{CuSO}_4$  solution, or even the powdered salt, to the solution of  $\text{KHCO}_3$ . Sulphates exert no bad effect on the reagent, as might be inferred from the instruc-

tions of some to wash the copper carbonate precipitate. Experiments have shown this not to be so, and that 0.0005 gramme glucose dissolved in 10 c.c. can easily be detected when this is boiled for ten minutes with an equal volume of the reagent. The copper separates as intensely red suboxide. Under the above conditions less than the given amount of glucose cannot be detected. If, however, the test is made in the following manner, then 0.00025 gramme of glucose can easily be detected. The concentrated solution of the latter is mixed with just enough reagent to colour it slightly, and evaporated to dryness on the water bath. If glucose is present the porcelain dish will show a rose-coloured residue, which in the absence of glucose would be bluish green. Addition of water will show the colour more distinctly. To avoid mistakes, it is well to make a blank with the reagent similarly diluted.

The quantitative determination with this reagent  $\left(\frac{N}{10}\right)$  must be made as follows: Measure out 100 c.c. (corresponding to 10 c.c. of a solution containing 0.05 gramme pure glucose), heat until boiling, and add the unknown solution slowly until all the copper is reduced. When the approximate number of c.c. of the sugar solution required is thus determined the final estimation is made by boiling 100 c.c. of the reagent for five minutes in a 200 c.c. flask, adding the required volume of sugar solution from a burette, boiling fifteen minutes, and filtering after the liquid has been cooled rapidly. The filtrate should contain neither glucose nor copper.

This method gives such rapid and satisfactory results that Ettore Soldaini recommends the use of this  $\frac{N}{10}$  copper solution.

3. *Scheller's modifications.*—Scheller states that Soldaini's reagent can be made of a definite composition by dissolving 15.8 grammes pure copper sulphate and 7.2 grammes potassium hydrate respectively in 100 c.c. of water, mixing and adding to 1700 c.c. of a solution of 594 grammes  $K_2CO_3$ . It is then heated on the water bath until the precipitate dissolves, and after cooling made up to 2000 c.c. 1 c.c. of this reagent contains 2 milligrammes of copper. It is said to be constant and stable.

4. *Preuss's researches.*—Preuss emphasises the necessity, in determining the reducing power of invert sugar with Soldaini's reagent, of always using a reducing liquor of constant composition, i.e., having a certain percentage of copper and degree of alkalinity. Scheller's method recommends itself by its simplicity. It consists in adding 15.8 grammes  $CuSO_4 \cdot 5H_2O$  to a hot solution of 594 grammes  $K_2CO_3$ , and making up to 2,000 c.c., when the precipitate is dissolved. Three solutions made thus showed the same density 1.1789, a similar reducing power to invert sugars, and were very stable. As it is to be expected that the advantages which Soldaini's reagent

possesses over Fehling's solution will cause its general adoption for the quantitative estimation of invert sugar, it will be of value to determine the exact reducing power of invert sugar with regard to this solution, especially as experiments so far made show it to be not exactly proportional to the quantity of invert sugar used. In the following experiments Soxhlet's 1 per cent. invert sugar solution was used, and the reduction made by heating 150 c.c. of the copper solution by the direct flame and adding the invert sugar from a pipette.

Upon boiling five minutes, 50 milligrammes invert sugar precipitates on an average 138 milligrammes copper; upon heating ten minutes, 156 milligrammes copper:

Period boiled.	Mg. Copper precipitated.
5 . . . . .	138
10 . . . . .	156
15 . . . . .	162
75 . . . . .	190
15 . . . . .	75 mg. Invert Sugar. 231
5 . . . . .	25 mg. Invert Sugar. 72
10 . . . . .	83
15 . . . . .	93
5 . . . . .	40 mg. Invert Sugar. 112
10 . . . . .	127

This shows that a definite time of boiling must be observed in using Soldaini's reagent for quantitative purposes. As the separation of copper increases up to fifteen minutes' boiling, and, on the other hand, such long boiling renders analysis cumbersome and the solution of copper suboxide possible, ten minutes were taken as the basis for the determination of the curve of reduction. Under these conditions—

	Average.
I. 5 mg. invert sugar reduced . . .	19 mg. Cu.
II. 10 mg. invert sugar reduced . . .	34 mg. Cu.
III. 25 mg. invert sugar reduced . . .	83 mg. Cu.
IV. 40 mg. invert sugar reduced . . .	128 mg. Cu.
V. 50 mg. invert sugar reduced . . .	167 mg. Cu.
VI. 60 mg. invert sugar reduced . . .	184 mg. Cu.
VII. 70 mg. invert sugar reduced . . .	214 mg. Cu.

Using these values, the following proportion is calculated for the curve of reduction by the method of the least squares—

$$y = 2.2869 + 3.299x - 0.004101x^2,$$

where  $x$  represents the milligrammes invert sugar used, and  $y$  the corresponding amount of copper. By solving the quadratic equation the invert sugar can easily be calculated from the copper found. The following table has been prepared to facilitate this—

TABLE LXXI.—INVERT SUGAR GRAVIMETRIC DETERMINATION.

Mg. Invert Sugar.	Mg. Cu.	Mg. Invert Sugar.	Mg. Cu.	Mg. Invert Sugar.	Mg. Cu.	Mg. Invert Sugar.	Mg. Cu.
5	18.8	18	60.3	81	100.6	44	139.5
6	21.9	19	63.5	32	103.6	45	142.4
7	25.2	20	66.6	33	106.6	46	145.4
8	28.4	21	69.7	34	109.7	47	148.3
9	31.6	22	72.9	35	112.7	48	151.2
10	34.9	23	76.0	36	115.7	49	154.1
11	38.1	24	79.1	37	118.7	50	157.0
12	41.3	25	82.2	38	121.8	51	171.3
13	44.5	26	85.3	39	124.8	52	185.5
14	47.7	27	88.5	40	127.8	53	200.4
15	50.9	28	91.4	41	130.7	54	213.1
16	54.0	29	94.5	42	133.6	55	226.6
17	57.2	30	97.6	43	136.5	56	240.0

The values of the table correspond satisfactorily to the quantities of copper found experimentally, as the following shows:—

TABLE LXXII.—INVERT SUGAR GRAVIMETRIC DETERMINATION.

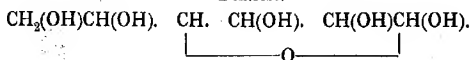
Mg. Invert Sugar.	Mg. Copper.	
	Calculated	By experiment.
5	18.8	19
10	4.9	34
25	82.2	88
40	127.8	128
50	157.0	157
60	185.5	184
70	213.1	214

In carrying out the estimations of invert sugar by Soldaini's reagent, we should closely follow the method of manipulation laid down, using for reduction an amount of polarised filtrate corresponding to ten grammes of substance. The filtration must not be too slow, and too great a dilution must be avoided, so that no subsequent formation of copper oxyhydrate or cuprous oxide takes place.

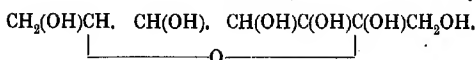
*Wilhelmy's researches.*—The time element in inversion has been studied by Wilhelmy and others. The invertive power of very small quantities of mineral acids in concentrated sugar solutions is pointed out, especially with a mere trace of HCl, which, when melted with sucrose, secures a complete inversion thereof, and that too without noticeable discolouration. Illustrations are given of the action of HCl on concentrated sugar solutions. 13.024 grammes of

sucrose were heated for thirty minutes in a stoppered flask at  $100^{\circ}$ , and then showed a polarisation of  $12^{\circ}$ . With longer heating the left-handed polarisation diminished, and also the quantity of copper precipitated. It is concluded from the diminution of the left-handed polarising power of the invert sugar, caused by concentration of the solution and of the acid, and by high temperature and duration of the invertive process, that the invert sugar formed is not pure levulose and dextrose, but also contains a different sugar. If this diminution be due to a molecular condensation of the invert sugar formed, then it would take place also when pure invert sugar is heated with an acid. This experiment was tried with invert sugar and hydrochloric acid. Pure dextrose and levulose were used for these experiments. 3.425 grammes of each were taken and made up to 50 c.c. at  $20^{\circ}$ , polarising  $15^{\circ}8$ . 0.1714 gramme of the mixed sugars gave 0.313 gramme copper. On heating this, as before, with dilute HCl, both polarisation and reduction were diminished. Heating with water alone gave no change, so that the condensation of the molecules of invert sugar is due to heating with acids. It was necessary, in order to elucidate the problem further, to note the action of HCl on concentrated glucose and levulose solutions. These results showed that the dextrose was but little affected by the treatment, and hence that the changes noted had been due to polymerisation of the levulose. An examination of the products formed by the action of a small quantity of acid on concentrated levulose solutions shows that dextrin-like (levulin-like) bodies are formed (levulosin). An 80 per cent. solution of dextrose was heated at  $105^{\circ}$  with increasing quantities of acid. This treatment increased the polarisation of the substance, and diminished its reducing power, indicating a polymerisation and formation of dextrinoids. Whence it follows that the hydrolytic splitting up of the di- and poly-saccharides is not a simple process, but together with the inverting action of the acid shown in the splitting up of the higher molecular weights, there is synchronous reformation of the polymers. These phenomena give support to the opinion of Tollens, that the monoses are not penta-oxaldehydes or penta-oxketones, but penta-oxoxides, that is, aldehyd or keto- $\gamma$ -lactones, in which the structural formulæ for dextrose and levulose are as follows:—

Dextrose.



Levulose.



It is further shown that the usual methods of inversion use, without exception, excessive quantities of acid, and that the maximum levogyrous result and maximum reducing power are to be obtained only with very dilute acids or at low temperatures. In order to convert pure sugar into colourless invert sugar, it is only necessary to heat the solution at 95° for half an hour with 0.01 per cent. HCl. The above principles of inversion were also applied to starch, inulin, etc., on the supposition that the difficulty of securing a complete inversion of starch lay in the reformation of dextrine by the strong acid employed, rather than in the natural refractory nature of the material, and experiments are given supporting this hypothesis. In the usual methods of inversion excessive quantities of acids are invariably used. The maximum levogyrous result and maximum reducing power are to be obtained only with very dilute acids or at low temperatures. To convert pure sugar into colourless invert sugar, it is only necessary to heat the solution at 95° C. for a half-hour with 0.1 per cent. HCl. That the difficulty in securing a complete inversion of starch lies in the reformation of dextrin by the strong acid employed, rather than in the natural refractory nature of the material, has been demonstrated by experiment.

*Maumené's theory.*—According to Maumené, invert sugar, even when inverted by pure water, is never a mixture of equal weights of glucose and levulose, or even of chylarose. Glucose is a mixture of several hexeloses; one of these, left-handed hexelose, approaches very near in its composition to what is called levulose derived from inulin. It is very difficult to isolate it, even at a freezing temperature, and it is unstable in a higher temperature. Another optically neuter, which has been called *mactose*, is obtained by a complete transformation of normal sugar by means of nitrate of silver. The third is a non-fermentable hexelose. It occurs in the inversion of normal sugar by pure water, or even by water which has been treated by a few ten-thousandths of acid. It is separated from the others by fermentation. The character of invert sugar is influenced: (1) By the relative quantities of sugar, water and acid; (2) by the degree of heat; (3) by the time of the reaction; (4) temperature during the subsequent treatment with lime, saturation of the product by carbonic acid, and the duration of the evaporation *in vacuo*. Inversion is produced by a series of molecular movements modified by these numerous influences, and the greatest precautions are necessary to obtain the same result in like conditions. Glucose retains a certain quantity of a mixture of hexeloses, properly named *chylarose*, since it is extracted from the invert sugar by capillary attraction, porous brick or porcelain, or blotting paper. According to Maumené, Jungfleisch and Grimbart have confirmed the alteration of left-handed hexelose, not upon this hexelose properly so called, but on the inverted sugar, by a comparison with a product of

inulin, a product which cannot be assimilated to invert sugar without many errors. The conclusions to which Jungfleisch and Grimbert came as the results of the experiments, to which Maumené takes exception, were: (1) Strong acids modify the rotatory power of levulose, by increasing it more or less according to circumstances; (2) the levulose of sugar inverted by the processes in ordinary use is not strictly identical with pure crystallised levulose.

*Jungfleisch and Grimbert's researches.*—Some facts which have been developed in the experience of the authors lead them to doubt the principle of the method of Clerget. The process of Clerget may be expressed as follows: Let  $A$  be the deviation produced by 1 decimetre of the liquor in which it is desired to find the weights  $x$  and  $y$  of the sucrose and of the inverted sugar. Let  $A'$  represent the deviation of the same liquid inverted by strong acid without changing its concentration; then  $(a)D = 67.31$  and  $(a)D = -24.31$ , this being the expression for the rotatory power of the sucrose and inverted sugar; then the two equations which will give the value of  $x$  and  $y$  are as follows—

$$\frac{67.31}{100}x - \frac{24.31}{100}y = A; \text{ and } \frac{24.31}{100}(x+y) = A'.$$

The results obtained in this way are generally inexact. Contrary to what is supposed in the first equation, inverted sugar exists in natural sugar products which have not been subjected to the action of strong acids; and its rotatory power, which at first is about  $(a)D = 21.16$ , increases greatly during inversion. Therefore the calculation indicates a plus error for the second, an error proportional to  $\frac{y}{x}$ , this increasing with the weight of inverted sugar pre-existing; moreover, the calculation exaggerates the error committed. It is known that if such a sugar be treated with strong and dilute acids the product is always more strongly left-handed in the first than in the second case. In the analysis of the crystallised sugar of commerce, which is always poor in inverted sugar, the difference is very little, and may be neglected; but such is not the case with sugars rich in inverted sugar. When the substances analysed contain glucose and levulose in unequal quantities, the other compounds are sometimes determined by the aid of an analogous calculation by estimating a third equation from the reduction of alkaline copper solution, but the same objection still applies. Acetic acid acts upon saccharose without modifying the levulose, but with some salts certain facts prevent its general use, namely: (1) Alkaline acetates, although they do not prevent the inversion of sucrose by strong acids employed in excess, interfere with the inversion by acetic acid even when employed in very great excess. In



presence of 1 molecule of acetate of soda 1 molecule of sucrose is not completely converted, even at  $100^{\circ}$ , during half an hour by a weight of acetic acid equal to 80 molecules. (2) Alkaline citrates, formiates, lactates and tartrates, the acetates of zinc and lead, act in a similar manner. The acetate of lime is much less active. (3) The salts of strong monobasic acids do not prevent inversion by acetic acid. The neutral salts of strong bibasic acetates diminish the inverting power when the base is monovalent, but not when the base is bivalent. The acid salts of strongly polybasic acids do not retard the inversion by acetic acid, but sometimes even affect it themselves.

*Gravimetric analysis of glucose.*—*Soxhlet's gravimetric method.*—It is important to always work under the same conditions: 60 c.c. of Fehling's solution, which does not require to be titrated, and which may be prepared there and then, is mixed with 60 c.c. of water. It is boiled, and all at once 25 c.c. of a solution, containing at the most 4 per cent. of sugar, is added, and after two minutes' boiling it is filtered through a special funnel of the same shape as the old calcium chloride tubes, 12 centimetres long and 13 millimetres in diameter, the bulb of which is half-filled with asbestos fibres, not too soft, and laid crosswise as far as possible. The asbestos is washed with boiling water, dried, and the tube weighed exactly. It is then put in communication with an aspirating flask, and, with the aid of a funnel, the liquid with the precipitate is poured into the tube. The precipitate is first washed with boiling water, then with alcohol, then twice with absolute alcohol, and finally twice with ether. The ether is then driven off by means of a current of air. The lower orifice of the filtration tube is then connected with a hydrogen-generating apparatus, the tube is inclined, and the bulb is heated by a small flame, care being taken to keep the point of it 5 centimetres below the bulb. In two or three minutes the reduction of the oxide of copper is complete. The tube is cooled in a current of hydrogen, a current of air is then passed through it, and the tube with its contents weighed. The quantity of sugar may be calculated from the following tables. (See also notes on p. 383):—

TABLE LXXIII.—GRAVIMETRIC ESTIMATION OF REDUCING SUGARS.

Mg. Cu.	Mg. Glucose.	Mg. Cu.	Mg. Glucose.	Mg. Cu.	Mg. Glucose.
10	6.1	50	25.9	300	156.5
20	11.0	100	50.9	400	212.9
30	16.0	200	102.6	463	249.9



TABLE LXXV.—ESTIMATION OF INVERT SUGAR, CALCULATED FROM E. MEISSEL'S DATA BY E. WEIN.

Mg. Copper.	Mg. Invert Sugar.	Mg. Copper.	Mg. Invert Sugar.	Mg. Copper.	Mg. Invert Sugar.	Mg. Copper.	Mg. Invert Sugar.	Mg. Copper.	Mg. Invert Sugar.
90	46.9	135	70.8	180	95.2	225	120.4	270	146.1
91	47.4	136	71.3	181	95.7	226	120.9	271	146.7
92	47.9	137	71.9	182	96.2	227	121.5	272	147.2
93	48.4	138	72.4	183	96.8	228	122.1	273	147.8
94	48.9	139	72.9	184	97.3	229	122.6	274	148.4
95	49.5	140	73.5	185	97.8	230	123.2	275	149.0
96	50.0	141	74.0	186	98.4	231	123.8	276	149.5
97	50.5	142	74.5	187	99.0	232	124.3	277	150.1
98	51.1	143	75.1	188	99.5	233	124.9	278	150.7
99	51.6	144	75.6	189	100.1	234	125.5	279	151.3
100	52.1	145	76.1	190	100.6	235	126.0	280	151.9
101	52.7	146	76.7	191	101.2	236	126.6	281	152.5
102	53.2	147	77.2	192	101.7	237	127.2	282	153.1
103	53.7	148	77.8	193	102.3	238	127.8	283	153.7
104	54.3	149	78.3	194	102.9	239	128.3	284	154.3
105	54.8	150	78.9	195	103.4	240	128.9	285	154.9
106	55.3	151	79.4	196	104.0	241	129.5	286	155.5
107	55.9	152	80.0	197	104.6	242	130.0	287	156.1
108	56.4	153	80.5	198	105.1	243	130.6	288	156.7
109	56.9	154	81.0	199	105.7	244	131.2	289	157.2
110	57.5	155	81.6	200	106.3	245	131.8	290	157.8
111	58.0	156	82.1	201	106.8	246	132.3	291	158.4
112	58.5	157	82.7	202	107.4	247	132.9	292	159.0
113	59.1	158	83.2	203	107.9	248	133.5	293	159.6
114	59.6	159	83.8	204	108.5	249	134.1	294	160.2
115	60.1	160	84.3	205	109.1	250	134.6	295	160.8
116	60.7	161	84.8	206	109.6	251	135.2	296	161.4
117	61.2	162	85.4	207	110.2	252	135.8	297	162.0
118	61.7	163	85.9	208	110.8	253	136.3	298	162.6
119	62.3	164	86.5	209	111.3	254	136.9	299	163.2
120	62.8	165	87.0	210	111.9	255	137.5	300	163.8
121	63.3	166	87.6	211	112.5	256	138.1	301	164.4
122	63.9	167	88.1	212	113.0	257	138.6	302	165.0
123	64.4	168	88.6	213	113.6	258	139.2	303	165.6
124	64.9	169	89.2	214	114.2	259	139.8	304	166.2
125	65.5	170	89.7	215	114.7	260	140.4	305	166.8
126	66.0	171	90.3	216	115.3	261	140.9	306	167.3
127	66.5	172	90.8	217	115.8	262	141.5	307	167.9
128	67.1	173	91.4	218	116.4	263	142.1	308	168.5
129	67.6	174	91.9	219	117.0	264	142.7	309	169.1
130	68.1	175	92.4	220	117.5	265	143.2	310	169.7
131	68.7	176	93.0	221	118.1	266	143.8	311	170.3
132	69.2	177	93.5	222	118.7	267	144.4	312	170.9
133	69.7	178	94.1	223	119.2	268	144.9	313	171.5
134	70.3	179	94.6	224	119.8	269	145.5	314	172.1

TABLE LXXV.—ESTIMATION OF INVERT SUGAR—continued.

Mg. Copper.	Mg. Invert Sugar.	Mg. Copper.	Mg. Invert Sugar.	Mg. Copper.	Mg. Invert Sugar.	Mg. Copper.	Mg. Invert Sugar.	Mg. Copper.	Mg. Invert Sugar.
315	173.7	339	187.2	362	201.1	385	215.5	408	230.7
316	173.8	340	187.3	363	201.7	386	216.1	409	231.4
317	173.9	341	188.4	364	202.3	387	216.8	410	232.1
318	174.5	342	189.0	365	203.0	388	217.4	411	232.8
319	175.1	343	189.6	366	203.6	389	218.0	412	233.5
320	175.6	344	190.2	367	204.2	390	218.7	413	234.3
321	176.2	345	190.8	368	204.8	391	219.3	414	235.0
322	176.8	346	191.4	369	205.5	392	219.9	415	235.7
323	177.4	347	192.0	370	206.1	393	220.5	416	236.4
324	178.0	348	192.6	371	206.7	394	221.2	417	237.1
325	178.6	349	193.2	372	207.3	395	221.8	418	237.8
326	179.2	350	193.8	373	208.0	396	222.4	419	238.5
327	179.8	351	194.4	374	208.6	397	223.1	420	239.2
328	180.4	352	195.0	375	209.2	398	223.7	421	239.9
329	181.0	353	195.6	376	209.9	399	224.3	422	240.6
330	181.6	354	196.2	377	210.5	400	224.9	423	241.3
331	182.2	355	196.8	378	211.1	401	225.7	424	242.0
332	182.8	356	197.4	379	211.7	402	226.4	425	242.7
333	183.5	357	198.0	380	212.4	403	227.1	426	243.4
334	184.1	358	198.6	381	213.0	404	227.8	427	244.1
335	184.7	359	199.2	382	213.6	405	228.6	428	244.9
336	185.4	360	199.8	383	214.3	406	229.3	429	245.6
337	186.0	361	200.4	384	214.9	407	230.0	430	246.3
338	186.6								

*Notes to Tables LXXIV.-LXXVII: Solution A.*—Pure (commercially)  $\text{CuSO}_4$ , 5 aq is crystallised once from dil.  $\text{HNO}_3$  and three times from water transferred to filter paper, dried and exposed to air for 12 hours. Dissolve 69.278 grammes in water to 1 litre. *Solution B.*—173 grammes Rochelle salt dissolved in 400 c.c. water and made up to  $\frac{1}{2}$  litre with 100 c.c. caustic soda (516 g.  $\text{NaHO}$  to litre) fresh made for immediate addition to A. (1) Table LXXIV., Dextrose 30 c.c. A., 30 c.c. B., 60 c.c. water, are boiled and 25 c.c. sugar solution containing not more than 1 per cent. are added and boiled for two minutes. (2) Table LXXV., Invert sugar 25 c.c. A., 25 c.c. B., are mixed with the sugar solution (which should not contain more than 0.2458 invert sugar) diluted with 100 c.c. water and boiled two minutes.

*Inversion Method.*—Dissolve 20 grammes of sugar in 700 c.c. water, add 100 c.c. dil.  $\text{HCl}$  (0.72 per cent.  $\text{HCl}$ ), heat thirty minutes in water bath, cool, neutralise with standard soda, dilute to 1 litre (0.95 invert sugar = Sucrose). Table LXXVI., Maltose, 25 c.c. A., 25 c.c. B., with 25 c.c. of sugar solution containing not more than 1 per cent., boil four minutes. Table LXXVII., 25 c.c. A., 25 c.c. B., are mixed with 20 to 100 c.c. milk sugar according to concentration made up to 150 c.c. with water, boil six minutes.

[The precipitates are treated as directed on p. 380.]



# THE CHEMISTRY OF SUGARS

385

TABLE LXXVII.—TABLE FOR USE IN THE GRAVIMETRIC ESTIMATION OF MILK SUGAR.

Mg. Cu.	Mg. Milk Sugar.	Mg. Cu.	Mg. Milk Sugar.	Mg. Cu.	Mg. Milk Sugar.	Mg. Cu.	Mg. Milk Sugar.	Mg. Cu.	Mg. Milk Sugar.
100	71.6	145	105.1	190	139.3	235	173.1	280	208.3
101	72.4	146	105.8	191	140.0	236	173.9	281	209.1
102	73.1	147	106.6	192	140.8	237	174.6	282	209.9
103	73.8	148	107.3	193	141.6	238	175.4	283	210.7
104	74.6	149	108.1	194	142.3	239	176.2	284	211.5
105	75.3	150	108.8	195	143.1	240	176.9	285	212.3
106	76.1	151	109.6	196	143.9	241	177.7	286	213.1
107	76.8	152	110.3	197	144.6	242	178.5	287	213.9
108	77.6	153	111.1	198	145.4	243	179.3	288	214.7
109	78.3	154	111.9	199	146.2	244	180.1	289	215.5
110	79.0	155	112.6	200	146.9	245	180.8	290	216.3
111	79.8	156	113.4	201	147.7	246	181.6	291	217.1
112	80.5	157	114.1	202	148.5	247	182.4	292	217.9
113	81.3	158	114.9	203	149.2	248	183.2	293	218.7
114	82.0	159	115.6	204	150.0	249	184.0	294	219.5
115	82.7	160	116.4	205	150.7	250	184.8	295	220.3
116	83.5	161	117.1	206	151.5	251	185.5	296	221.1
117	84.2	162	117.9	207	152.2	252	186.3	297	221.9
118	85.0	163	118.6	208	153.0	253	187.1	298	222.7
119	85.7	164	119.4	209	153.7	254	187.9	299	223.5
120	86.4	165	120.2	210	154.5	255	188.7	300	224.4
121	87.2	166	120.9	211	155.2	256	189.4	301	225.2
122	87.9	167	121.7	212	156.0	257	190.2	302	225.9
123	88.7	168	122.4	213	156.7	258	191.0	303	226.7
124	89.4	169	123.2	214	157.5	259	191.8	304	227.5
125	90.1	170	123.9	215	158.2	260	192.5	305	228.3
126	90.9	171	124.7	216	159.0	261	193.3	306	229.1
127	91.6	172	125.5	217	159.7	262	194.1	307	229.8
128	92.4	173	126.2	218	160.4	263	194.9	308	230.6
129	93.1	174	127.0	219	161.2	264	195.7	309	231.4
130	93.8	175	127.8	220	161.9	265	196.4	310	232.2
131	94.6	176	128.5	221	162.7	266	197.2	311	232.9
132	95.3	177	129.3	222	163.4	267	198.0	312	233.7
133	96.1	178	130.1	223	164.2	268	198.8	313	234.5
134	96.9	179	130.8	224	164.9	269	199.5	314	235.3
135	97.6	180	131.6	225	165.7	270	200.3	315	236.1
136	98.3	181	132.4	226	166.4	271	201.1	316	236.8
137	99.1	182	133.1	227	167.2	272	201.9	317	237.6
138	99.8	183	133.9	228	167.9	273	202.7	318	238.4
139	100.5	184	134.7	229	168.6	274	203.5	319	239.2
140	101.3	185	135.4	230	169.4	275	204.3	320	240.0
141	102.0	186	136.2	231	170.1	276	205.1	321	240.7
142	102.8	187	137.0	232	170.9	277	205.9	322	241.5
143	103.5	188	137.7	233	171.6	278	206.7	323	242.3
144	104.3	189	138.5	234	172.4	279	207.5	324	243.1

TABLE LXXVII.—GRAVIMETRIC ESTIMATION OF MILK SUGAR—*continued*.

Mg. Cu.	Mg. Milk Sugar.	Mg. Cu.	Mg. Milk Sugar.	Mg. Cu.	Mg. Milk Sugar.	Mg. Cu.	Mg. Milk Sugar.	Mg. Cu.	Mg. Milk Sugar.
325	249.9	341	256.5	356	268.8	371	281.4	386	294.2
326	244.6	342	267.4	357	269.6	372	282.2	387	295.1
327	245.4	343	258.2	358	270.4	373	283.1	388	296.0
328	246.2	344	259.0	359	271.2	374	283.9	389	296.8
329	247.0	345	259.8	360	272.1	375	284.8	390	297.7
330	247.7	346	260.6	361	272.9	376	285.7	391	298.5
331	248.5	347	261.4	362	273.7	377	286.5	392	299.4
332	249.2	348	262.3	363	274.5	378	287.4	393	300.3
333	250.0	349	263.1	364	275.3	379	288.2	394	301.1
334	250.8	350	263.9	365	276.2	380	289.1	395	302.0
335	251.6	351	264.7	366	277.1	381	289.9	396	302.8
336	252.5	352	265.5	367	277.9	382	290.3	397	303.7
337	253.3	353	266.3	368	278.8	383	291.7	398	304.6
338	254.1	354	267.2	369	279.6	384	292.5	399	305.4
339	254.9	355	268.0	370	280.5	385	293.4	400	306.3
340	255.7								

TABLE LXXVIII.—GRAVIMETRIC ESTIMATION OF SMALL QUANTITIES OF GLUCOSE (HERZFELD).

Copper Mg.	Invert Sugar per Cent.	Copper Mg.	Invert Sugar per Cent.	Copper Mg.	Invert Sugar per Cent.
50	0.05	140	0.51	230	1.02
55	0.07	145	0.53	235	1.05
60	0.09	150	0.56	240	1.07
65	0.11	155	0.59	245	1.10
70	0.14	160	0.62	250	1.13
75	0.16	165	0.65	255	1.16
80	0.19	170	0.68	260	1.19
85	0.21	175	0.71	265	1.21
90	0.24	180	0.74	270	1.24
95	0.27	185	0.76	275	1.27
100	0.30	190	0.79	280	1.30
105	0.32	195	0.82	285	1.33
110	0.35	200	0.85	290	1.36
115	0.38	205	0.88	295	1.38
120	0.40	210	0.90	300	1.41
125	0.43	215	0.93	305	1.44
130	0.45	220	0.96	310	1.47
135	0.48	225	0.99	315	1.50

TABLE LXXIX.—COMPARISON OF VIOLETTE'S, FEHLING'S, KNAPP'S AND SACHS' SOLUTIONS.

Sugar.	Fehling's 10 c.c. =	Violette's 10 c.c. =	Soxhlet's Figures.		
			Fehling's 1 c.c. =	Knapp's <sup>1</sup> 1 c.c. =	Sachs' <sup>2</sup> 1 c.c. =
	Gramme.	Gramme.	Mg.	Mg.	Mg.
Sucrose . . .	0.0475	0.050	—	—	—
Dextrose . . .	0.048	0.051	4.758	2.010	3.305
Invert sugar . . .	0.050	0.053	4.911	1.990	2.660
Levulose . . .	0.052	0.055	5.144	1.970	2.225
Lactose . . .	0.067	0.072	6.757	3.100	4.660
Maltose . . .	0.075	0.079	7.780	3.100	5.060
Galactose . . .	—	—	5.110	2.420	4.420
Melazitose . . .	—	—	—	2.230	3.880

*Fehling's solution.*— $\text{CuSO}_4$ , 5Aq 34.64 grammes; Rochelle salt, 173 grammes; caustic soda solution, 480 c.c.; distilled water to 1 litre. *Test for cane sugar.*—2 c.c. of the sugar solution, 5 drops of a 20 per cent. alcoholic solution of *a*-naphthol, and 10 c.c. pure conc.  $\text{H}_2\text{SO}_4$  are mixed in a test-tube. The liquid is coloured violet or bright rose, according to the percentage of sugar. The reaction is very decided with 1 part of sugar in 100,000 of water, and even smaller quantities may be detected. Pellet has introduced a quantitative process based on this reaction. But it must not be forgotten that many carbohydrates give this reaction, and that it may even be used to detect the presence of a carbohydrate radicle in a proteid.

*Analysis of Raw Sugar.*—The commercial analysis of raw sugar comprises the following determinations: 1, density; 2, moisture; 3, ash; 4, cane sugar; 5, uncrystallisable sugar; 6, organic matter; 7, yield of refined sugar.

1. *Density and total solids.*—The density of juices, syrups, etc., is determined by means of Baumé's or Brix's hydrometer or areometer, preferably with the latter, as graduations of the scale give close approximations to the percentages of total solids. The Brix spindle should be graduated to tenths, the divisions of the scale being of sufficient length to enable the analyst to note exactly the point to which the stem sinks in the liquid. It is desirable that the range of degrees recorded by each individual spindle be as limited as possible, this end being best secured by the employment of sets, consisting of not less than three spindles. The solutions should be as nearly as possible of the same temperature as the air at the time of reading, and if the variation from the standard temperature of the graduation of the spindle amounts to more than  $1^\circ \text{C}$ ., compensation therefor must be made by reference to the table of corrections for temperature on page 340. The juice should be allowed to

<sup>1</sup>  $10\text{gHCl} + 100 \text{ c.c. NaHO}$ , solution of density 1.145 to 1 litre.

<sup>2</sup>  $18\text{gHgI}_2 + 25\text{gKI} + 50\text{gKHO}$ .



stand in the cylinder until all air-bubbles have escaped before reading the spindle. To accurately determine the specific gravity of the sample, a specific-gravity bottle or picnometer of 50 c.c. capacity is employed, and the weight of the amount required to fill the bottle at 15°5 C., divided by 50, gives its correct specific gravity. When the temperature varies from the standard temperature for specific gravities, the weight of the juice must be compared with an equal volume of water weighed at the same temperature. The per cent. of *total solids* can be approximately ascertained directly from the reading of the Brix hydrometer; or, knowing the reading of the Baumé spindle, or the specific gravity of the sample, by reference to the usual tables the desired percentage can be obtained. To accurately estimate the proportion of total solids in a juice or syrup, from 1 to 3 grammes (according to the density of the liquid) are run into a porcelain dish or glass schaelchen, containing about 2 grammes of finely powdered ignited silica. The whole is then placed on a water bath and heated until the contents are apparently dry, after which it is transferred to an air bath and dried to a constant weight at a temperature not exceeding 110° C. The weight of the dish and silica subtracted from the weight of the dish and contents after drying gives total solids.

2. *Estimation of moisture*.—Moisture is determined on 5 grammes of substance, which is dried at 105° C. until of constant weight—about two hours for normal sugar. The ash determination is often made on that used for the moisture, so as to avoid a fresh weighing of 5 grammes.

(1) *In sugar or melada*.—Weigh from 2 to 5 grammes into a flat dish (nickel or platinum) and dry at 100° C. for three hours; cool in desiccator and weigh; return to oven and dry for one hour. If on weighing there is only a slight change of weight, the process may be considered finished; otherwise the drying must be continued until loss of water in one hour is not greater than 0·20 per cent.

(2) *In honey or molasses*.—(a) Proceed as above, using only 1 to 2 grammes of substance.

(b) Place 2 to 3 grammes of substance in a flat dish holding from 30 to 50 metres; dissolve in 70 per cent. alcohol, using a small stirring rod, which is weighed with the dish. Add from weighing bottle 15 to 25 grammes pure dry sand; dry in oven at 70° to 80° C. until alcohol is nearly driven off; moisten thoroughly with 99 per cent. absolute alcohol; dry at 75° C. for thirty minutes, then at temperature of boiling water for an hour; repeat as above, deduct weight of added sand and make usual calculations.

3. *Ash*.—(a) Take 5 to 10 grammes of the material (sugar, molasses, honey) in platinum dish of 50 to 100 c.c. capacity; heat at 100° C. until water is expelled and then slowly until intumescence ceases. The dish is then placed in muffle and heated at low redness until a white ash is obtained. If the substance contains tin or other metal

capable of uniting with platinum a dish made of some other material must be used.

(b) *Soluble ash*.—Digest ash with water, filter on to Gooch crucible, wash with hot water, dry residue at  $100^{\circ}$  C. and weigh. Difference of weights equals soluble ash.

(c) *Estimation of true ash in sugars and syrups without sulphuric acid*.—Oxide of zinc is used, which has the following qualities to recommend it: It is white, infusible, forms only a single oxide, is easily obtained in impalpable powder, is not hygroscopic, and it yields its oxygen as easily as the oxide of copper in the presence of organic matters, and in a measure indefinitely, because the zinc, reduced to a metallic state, is reoxidised immediately, and is susceptible, therefore, of furnishing a new quantity of oxygen to the substance until its complete combustion. The operation is conducted as follows:—

In the case of sugar 5 grammes are weighed in a platinum dish, to which are added 0.05 gramme oxide of zinc. Incorporate thoroughly by dilute alcohol, and mixing, dry. The carbonising is then carried on with due precaution over a Bunsen burner. The mass, on melting, absorbs evenly the oxide of zinc. After the carbonising is completed the capsule is placed in a muffle, heated to low redness, and the incineration is completed after three-quarters of an hour, or an hour at most. The ash is weighed and the 50 milligrammes of oxide of zinc subtracted. The capsule should be dried in a dessicator and weighed as rapidly as possible, as the carbonate of potash present is highly hygroscopic.

For syrups the operation is carried on in the same manner, except sometimes it is preferable to weigh only 3 grammes instead of 5. The capsules of platinum do not seem to suffer anything from the presence of the oxide of zinc, but porcelain dishes can be used instead.

*Estimation of ash in sugar by incineration with benzoic acid*.—To avoid correction of  $\frac{1}{10}$  as proposed by Scheibler, and  $\frac{1}{5}$  as proposed by Girard and Violette, when sugars are burnt with  $\text{H}_2\text{SO}_4$ , Boyer suggests incineration with benzoic acid as giving the real quantity of mineral matter without correction. The benzoic acid is dissolved in 90 per cent, alcohol, 25 grammes of the acid to 100 c.c. of alcohol. Five grammes of the sugar are weighed in a capsule and moistened with 1 c.c. water. The capsule is heated slowly to caramelise the sugar without carbonising it; 2 c.c. of the benzoic solution are next added, and the capsule warmed until all the alcohol is evaporated; the temperature is then increased until the sugar is carbonised. The decomposing benzoic acid produces abundant vapours, which render the mass extremely porous, especially if a circular motion be applied to the capsule. The slow heating is continued until all benzoic acid is volatilised. The carbon obtained is voluminous and brilliantly black. The incineration is finished in a muffle at a low red heat. The capsule is weighed quickly when taken from dessi-

cator to avoid absorption of water by alkaline carbonates. Benzoate of ammonia may be used instead of benzoic acid, and freedom of both acid and ammonia salt from residue on ignition should be tested. Besides giving mineral matters directly, by this method their composition may also be determined—a rather important item.

(d) Carbonise the mass at a low heat; dissolve soluble salts with hot water; burn residual mass as above, add the solution of soluble salts and evaporate to dryness at  $100^{\circ}\text{C}$ .; ignite gently, cool in dessicator and weigh.

(e) Saturate sample with  $\text{H}_2\text{SO}_4$ , dry, ignite gently, then burn in muffle at low redness.

The increase in weight gives sulphated ash in 5 grammes of the sugar, which, multiplied by 20, brings it to per cent. As weight of ash rarely exceeds 0.2 gramme, Sidersky uses a small invariable weight of 4.8 grammes, which he uses in every weighing of ash, only the weights varying between 0.1 and 0.2 gramme according to the richness of the sugar in ash, have then to be added, and the operator need only trouble himself with these weights, as the 4.8 grammes remain constant.

*Determination of the true ash in products of cane-sugar manufactories, incinerated with sulphuric acid.*—The determination of the ash in products of sugar manufactories is easily made by Scheibler's method (sulphuric incineration). It was proposed in 1865, to circumvent inconveniences in direct incineration proposed by Pelegot in 1851, and carbonisation with titration of aqueous extract of ash proposed by Dubrunfaut in 1832. Scheibler, recognising that sulphuric incineration gave a greater weight of ash than direct incineration, and that this difference might be calculated, applies to the sulphated ash a correction coefficient 0.90, now generally adopted. However, in 1870 Dubrunfaut drew attention to the fact this coefficient of 0.90 was only a mean value, which might vary sensibly from the truth in many cases. He then proposed to add to the sugars finely powdered baker's charcoal, or, better, platinum black. Dubrunfaut gives the following experiment. A sugar incinerated with spongy platinum gave 1.724 per cent. of ash; by direct incineration, 1.730; by sulphuric incineration, corrected by the coefficient 0.9, it gave 1.791. The first two results agree, giving a mean of 1.727, while in the third result the quantity obtained from a weight of sulphated

ashes of  $\frac{1.791}{0.9} = 1.99$ . A true coefficient to convert the sulphated

into true ash would thus be  $\frac{1.727}{1.99} = 0.868$ , and the coefficient 0.90 would be too high. This was Violette's conclusion, who suggested 0.8 instead of 0.9 as being more exact for raw sugars (excepting "firsts") for molasses, diffusion liquors, and the beet itself. For "firsts" sugars the coefficient should not be more than 0.7.

(f) Dissolve 10 grams of sugar in hot water filter to remove sand, etc.; evaporate filtrate and washings to dryness; carefully carbonise and extract with hot water until no longer any reaction for chlorine. Dry and ignite residue; weigh as insoluble ash. Add soluble portion, treat with HCl in slight excess, and evaporate to dryness. Heat until all free HCl is driven off; take up with  $H_2O$  and a little HCl, filter and wash. Residue is silica. Add  $NH_4OH$  to filtrate, boil and filter, wash residue, which is iron and alumina. To filtrate add  $(NH_4)_2C_2O_4$ , evaporate to dryness; ignite and moisten with  $(NH_4)_2CO_3$  and re-ignite; dissolve in water, filter and wash. Residue equals carbonates of Ca and Mg. Evaporate filtrate to small bulk, and  $(NH_4)_2CO_3$ , evaporate to dryness; drive off excess of ammonia cautiously, weigh. This gives alkalis as carbonates. This weight added to insoluble ash, gives total carbonate ash.

4. *Sucrose*.—The optical method is used when the sugar is approximately pure. The amount of sugar corresponding to the saccharimeter to be used—namely, 16.19 grammes for the French instrument and 26.048 for the German instrument—is dissolved in 50 to 70 c.c. of water. The sugar may be dissolved by weighing it in a platinum basin and dissolving it in the same dish; or it may be transferred to a test-glass on foot, the basin being rinsed with a little water, afterwards used to dissolve the sugar in the glass. The lumps of sugar difficult to dissolve are crushed with a glass rod (dubbed out into a knob at the end), also used to stir the liquid, and thus facilitate solution. Solution accomplished, the liquid is cautiously run into a 100 c.c. flask, the glass rod being placed against lip of platinum dish, or test-glass on foot, during operation, which with rod are washed with water to remove all trace of sugar. The wash waters are collected in the 100 c.c. flask so as to fill it to three-quarters. Some chemists prefer to operate in a different way. The sugar is weighed on a small gutter of sheet-aluminium, then the sugar is run directly through a nickel silver funnel into the 100 c.c. flask by the aid of a jet of water, which also rinses the funnel. When the sugar is greasy it often adheres to the aluminium foil used in weighing. The adherent crystals are removed by a flat brush made by trimming a goose quill, and the aluminium foil remains dry and ready for use immediately afterwards for the next experiment. The flask is then three-quarters filled by addition of water, after which a rotary movement is imparted to it to dissolve the sugar. Transferring and washing are thus avoided, an advantage when a series of similar analyses are to be made. But solution is effected more slowly. The sugar being completely dissolved, a little basic acetate of lead is added, 0.1 to 1 c.c., according to the depth of colour of the solution, to precipitate colouring matters, and water is then added to fill the flask almost to the mark, air-bells dispersed with a drop of ether, and the solution then made up to the mark

with water. The turbid liquid is shaken to render it homogeneous, filtered, and examined directly in the polarimeter for the percentage of saccharose in the raw sugar. In France polarimeters of the Laurent type are used, the normal official weight of which is 16.19 for 100 c.c. of the metric system, whereas in Belgium the German instruments of the Ventzke type are used, the normal weight of which is 26.048 grammes for 100 c.c. of Mohr's system (say 26 grammes for 100 c.c. metric system, see p. 354).

*U.S.A. official method.*—(a) Dissolve 26.048 (for the Ventzke), 16.19 (for the Laurent scale) grammes of sugar in water at temperature at which polarisation is to be made, and, after clarification, make volume up to 100 c.c. The sugar is conveniently weighed in a counterpoised nickel or German silver dish with large lip, whence it is readily washed into the flask. The solution before being filled to the mark is clarified by addition of lead subacetate or phosphotungstic acid until no further precipitation takes place. Excess of lead should be avoided. Filtration and clarification may often be helped by adding a few drops of suspended aluminic hydrate after the lead. The flask is filled with pure water until the lower line of meniscus coincides with mark on neck. The mouth of flask is closed with thumb, and its contents well shaken, and poured into a folded filter; the first 15 to 20 c.c. of filtrate are rejected. The subsequent filtrate, if not perfectly clear, is returned to filter, and this continued until filtrate shows no cloudiness. A 200 (100 to 500) mm. tube is filled with solution, placed in polariscope, the scale of which, after neutralising rotation produced by the sugar by turning the analytical prism or moving the quartz wedge, will give percentage of sucrose in sample taken. A dark solution may either be read in a 100-mm. tube, or it may be decolorised by shaking it with a small quantity of dry bone black. A weak solution may be read in a tube from 300 to 500 mm. in length.

(b) *Optical method by inversion.*—For raw sugars, molasses, etc.

(1) *Method of Clerget.*—Make up solution as above and place 50 c.c. of filtrate in a flask marked at 50 and 55 c.c. Fill to upper mark with strong HCl and mix well; place in hot water and heat quickly to 68° C.; keep at this temperature for exactly ten minutes; remove, cool quickly to room temperature, and polarise, noting temperature. If sample contained originally any invert sugar the second polarisation should be made at approximately the same temperature as the first. The percentage of sucrose is then calculated by the following formula:  $S$  = percentage of sucrose;  $a$  = first polarisation;  $b$  = second polarisation (usually to the left);  $a + b$  = sum of the polarisations;  $t$  = temperature of observation in degrees C.

$$\text{then } S = \frac{a + b}{144 - \frac{t}{2}}$$

When  $b$  is to left,  $a$  and  $b$  are to be added; when  $b$  is to right, it is to be subtracted from  $a$ . (2) *Method of Lintdet.*—Place 50 c.c. of filtrate as above in a flask marked at 100.5 c.c.; add 5 grammes of powdered zinc (zinc dust), place in boiling water, and when at boiling-point add, little by little, 5 c.c. of strongest HCl. After acid has all been added, cool and make up to mark. Polarise in a 400-mm. tube, or multiply reading in a 200-mm. tube by 2. Calculate percentage of sucrose as before. *Gravimetric method.*—Determine first any reducing sugar in sample by methods mentioned above; then invert sucrose, neutralise free acid, and redetermine reducing sugar. Deduct percentage of reducing sugar obtained at first, and remainder will be reducing sugar derived from sucrose; multiply this number by 95 to obtain percentage of sucrose in sample.

5. *Uncrystallisable sugar or reducing sugar.*—The presence of this sugar is confirmed by a qualitative test with Fehling's solution; in which case the liquid remaining from the polarimetric examination is freed from its excess of lead by mixing it with 5 c.c. of a saturated solution of carbonate or sulphate of soda, and filtering. The dilution of the liquid may be avoided by adding finely powdered dry sulphate of soda. The filtered liquid is run into a burette and  $\frac{1}{2}$  to 1 c.c. of Fehling's solution titrated therewith until the liquid floating on the precipitate of cuprous oxide becomes colourless. By titrating 1 c.c. of Fehling's solution, corresponding to 0.005 gramme of glucose, the quantity of glucose in 100 c.c. of the sugar solution ( $x$ ) is first found, then that in 100 grammes of the sugar ( $y$ ) by the following proportions:—

$$0.005 : n = x : 100; \quad x = \frac{0.5}{n};$$

$$\frac{0.5}{n} : \text{normal weight} = y : 100;$$

$$y = \frac{50}{n \times \text{normal weight}}.$$

In Germany the gravimetric method is used, weighing the metallic copper obtained by the reduction of the protoxide. In the case of sugars containing more than  $1\frac{1}{2}$  per cent. of glucose, Meissl's table (pp. 382 and 408) is used, whilst Herzfeld's table (p. 386) is used for those which contain less, always working on 10 grammes of sugar dissolved in 50 c.c. of liquid. So as to utilise the solution remaining from the polarisation (26.048 grammes in 100 c.c.), 76.8 c.c. of the liquid is drawn by means of a special pipette and a saturated solution of carbonate of soda added, and the bulk brought to 100 c.c. It is filtered and 50 c.c. of liquid drawn off, containing exactly 10 grammes of sugar.

The official U.S.A. method is as follows: *Reagent, alkaline copper*

*solution.* (*Fehling, Violette*) 34.64 grs. of pure crystallised copper sulphate. 187.00 grs. of tartrate of soda and potash. 78.00 grs. of caustic of soda. Dissolve the  $\text{CuSO}_4 + 5\text{H}_2\text{O}$  in water, and make up to 1,000 c.c. at the average temperature at which the analyses are to be made. Mix the solutions of Rochelle salts and caustic soda, and make up to 1,000 c.c. In practice use equal volumes of each reagent.

*Processes (volumetric).*—(a) The solution to be examined should contain about 1 per cent. of reducing sugar. To 10 c.c. of copper solution and 10 c.c. of alkali, in a large test tube or casserole, add 20 c.c. of water and boil. Add sugar solution in approximately proper quantity to reduce copper, and boil for exactly two minutes. If, on standing for a moment, the solution is still blue, add more sugar solution and re-boil. When no longer able to distinguish any residual copper by the eye, suck a few drops of the boiling hot liquid into a Wiley-Knorr end reaction tube, and test the filtrate for Cu with ferrocyanide of potassium solution and acetic acid. If preferred the Ross method, using filter paper, may be used.

A small filter, the diameter of which need not exceed 2 inches, is folded as for ordinary filtrations, and immersed, point down, in the hot supernatant liquid, the apex of the filter being just beneath the surface of the solution. A portion of the liquid at once filters through, and is removed from interior of filter by a small dropping pipette, consisting of a small glass tube with finely drawn out point and provided with a rubber bulb at the upper extremity. The liquid is readily taken up and expelled from the pipette by alternately compressing and relaxing pressure upon the bulb. A fresh filter is used for each filtration, and the pipette is rinsed after each portion of solution is tested. If copper be still present, add sugar solution little by little, boil, and re-test until no copper is found. Having thus determined approximate quantity of sugar solution to be used, repeat test twice, adding immediately each time almost the entire quantity of sugar solution necessary to complete reduction.

(b) *Gravimetric.*—Take 25 c.c. of copper solution of the following composition: 34.639 grs. of  $\text{CuSO}_4 + 5\text{H}_2\text{O}$  in 500 c.c. of water, add in an Erlenmeyer flask 25 c.c. of alkali solution made as follows: 173 grs. of Rochelle salts and 51.7 grs. of NaHO in 500 c.c. of water.

Add 50 c.c. of a sugar solution of such strength as to leave a part of the copper unreduced; heat quickly to boiling point, and boil for exactly two minutes; add 100 c.c. of recently boiled, cold, distilled water. The liquid is poured at once into a hard glass filter tube with asbestos diaphragm (an ordinary extraction tube answers well). The asbestos film should be previously moistened. Bring finally all separated suboxide into the tube by a feather brush, and wash with 300 to 400 c.c. of boiling water, afterward with 20 c.c. of absolute alcohol, and finally with ether; then dry, heat to low redness to convert into oxide, and to destroy any organic matter. The copper may also be

weighed in metallic state after reduction in stream of dry hydrogen. The tube should in all cases be kept in a desiccator before weighing.

(c) (*Gravimetric*).

Reagents:

34.639 grs. of $\text{CuSO}_4 + 5\text{H}_2\text{O}$	} in 500 c.c. of water.
125.000 grs. of $\text{NaOH}$	
173.000 grs. of Rochelle salts	

*Process*.—In a beaker of 250 c.c. capacity place 25 c.c. each of the reagents, and 50 c.c. water. Heat to boiling add 25 c.c. of the solution to be tested, which should contain about 1 per cent. of reducing sugar (dextrose, levulose), boil exactly two minutes, decant into Gooch crucible, wash by decantation with 100 c.c. of boiling-hot water keeping the cuprous oxide in beaker constantly covered. Place beaker containing cuprous oxide in place of the one receiving the wash water and dissolve  $\text{Cu}_2\text{O}$  on Gooch crucible in a fine stream of  $\text{HNO}_3$ ; wash until all  $\text{Cu}(\text{NO}_3)_2$  is removed. Transfer the whole of the nitrate from beaker to tared platinum dish, add a little  $\text{H}_2\text{SO}_4$ , evaporate over a steam bath until all  $\text{HNO}_3$  is expelled, and the Cu is converted into  $\text{CuSO}_4$ ; connect with battery of four gravity cells, and allow electrolytic action to continue for three hours, or until all Cu is deposited. (Connect dish with zinc pole of battery.) Pour off acid solution, adding water until every trace of  $\text{H}_2\text{SO}_4$  is removed; wash with 95 per cent. alcohol, and finally with absolute alcohol; ignite residual alcohol, cool in a desiccator, and weigh; calculate sugar reduced from weight of metallic copper by following factors.  
 (1) Multiply weight of copper by 0.5698 for weight of invert sugar.  
 (2) Multiply weight of copper by 0.5808 for weight of anhydrous dextrose. These factors are correct only for a copper solution of above strength and reducing sugar of approximately 1 per cent. strength.

6. *Organic matter* is estimated by difference.

7. *Yield of refined sugar*.—This is estimated in Britain by deducting from the polarimetric sugar the glucose multiplied by 3 and the ash by 5. In France the coefficients 2 and 4 are used, i.e., the uncrystallisable is multiplied by 2 and the ash by 4. Thus over there a sugar testing 95 per cent. by the polarimeter containing 0.1 per cent. glucose would have as refinery yield  $95 - 0.5 \times 2 - 1.5 \times 4 = 88$  per cent., and it is on this 88 per cent. that the tax is levied. The skill of the refiner is, therefore, to extract more than 88 per cent. of white sugar from the raw sugar he buys. This he easily does and gains the difference.

*Analysis of molasses*.—Molasses or treacle is the liquid, uncrystallisable, viscous mother-liquid from which sugar has been crystallised. Molasses from cane sugar contains about 35 per cent. of saccharose (crystallisable sugar), whilst that from beetroot contains 50 per cent. These relatively large amounts of real sugar cannot be crystallised,



on account of the great concentration of impurities by the elimination of several previous crops of sugar crystals from the liquid in question. Moreover, it requires the presence of a large quantity of water to keep these impurities in solution; so much so that any attempts to further concentrate the mass cause it to become so thick and consistent, that, even if it were possible for sugar to crystallise in such thick sticky media, it would be impossible to separate the crystals from such a viscous mother-liquid.

Gill gives the following analyses of molasses from both sources:—

TABLE LXXX.—CHEMICAL COMPOSITION OF MOLASSES.<sup>1</sup>

	Cane Molasses.	Beet Molasses.
	Per Cent.	Per Cent.
Cane sugar . . . . .	35.0	49.0
Glucose . . . . .	32.0	3.0
Ash . . . . .	5.5	12.5
Organic matter other than sugar (gum, etc.) . . . . .	9.5	15.5
Water . . . . .	18.0	20.0
	100.0	100.0

Cane molasses is much superior to beet molasses. From about 20 to 35 per cent. of the impurities present in cane sugar consist of sugar which has changed during the process. The sweetening capacity of this sugar is almost equal to that of crystallised cane sugar. It consequently commands a certain value on the market. But beet molasses has a nauseous bitter taste, due to saline impurities.

A glance at the above analyses suffices to show that beet molasses is highly charged with impurities. All impurities in the beet are concentrated in it with the impurities present in all the ingredients used in its treatment, such as those in the lime and the water, both of which deposit their soluble salts in it. Beet molasses is a highly complex organic and inorganic product of a gluey nature, a fusty smell, and very bad taste.

*Determining the density of molasses by the hydrometer.*—Owing to its great viscosity, it is not easy to determine the density of molasses. Baumé's or any other hydrometer only gives approximate results, and can only be used for getting some rough idea there-  
anent.

*By a 50 c.c. flask.*—A more exact method is to fill the whole of

<sup>1</sup> See also Tables LXXXII. and LXXXIII., p. 408, and LXXXIV., p. 404.

the bulb portion of a previously tared 50 c.c. flask with syrup, by running it in through a funnel with a long stem; it is then weighed to ascertain the weight of the molasses. The flask is then placed under a Mohr's burette containing water, and the flask is filled to the mark with water from the burette. The volume of water added is deducted from 50 c.c., and the difference represents the volume occupied by the molasses, the weight of which is known. The two elements for calculating the density are thus ascertained:

$$D = \frac{W}{V}.$$

*Analysis of molasses.*—The analysis of molasses is troublesome. If dissolved in water it yields a black liquid difficult to decolorise, entailing many precautions. Yet its analysis is necessary, because it contains on an average 50 per cent. of sugar, and it is sold by its percentage of the latter ingredient to the distiller; and it is by its analysis that the distiller is guided in his treatment of it, so as to convert it into alcohol.

Five times the weight of the quantity of molasses to be used in the saccharimeter employed—say 130·24 grammes for the German instrument and 81 grammes for the French instrument—are weighed into a capacious nickel capsule with a wide spout. Weighing is facilitated by the use of a glass rod, so as to add or remove a small drop of molasses as may be required. A small quantity of hot water is run into the capsule, stirring the mass with a glass rod. The molasses partially dissolves. The liquid is decanted into a graduated flask of 500 c.c. capacity, supporting the beak of the capsule against the mouth of the flask to avoid using a funnel. Hot water is again added to the remainder of the molasses to dissolve a fresh portion, which is likewise poured into the flask, and so on until the whole of the molasses weighed out is dissolved. The solution will then run to about 300 c.c. Towards the end the capsule is rinsed out with a jet of hot water, and the wash water added to the main solution. The whole is well agitated by rotating the flask, and brought to the surrounding temperature; 25 c.c. of basic acetate of lead are then added—that is to say, the volume strictly necessary to produce the precipitate. The volume is made up to 500 c.c., agitated and filtered, and 100 c.c. polarised. Should the filtered liquid be too dark to be exactly observed in the saccharimeter, it is decolorised with a little fine, dry, specially prepared animal charcoal, previously exhausted with hydrochloric acid exactly neutralised with alkali, washed and dried. The black is introduced into the filtered liquid with a little zinc dust, stirred with a rod, and filtered after fifteen or twenty minutes. The filtered liquid is then so far decolorised that it may be polarised. A confirmatory test is made by Fehling's solution. Possibly the indications of the saccharimeter

are not always exact, due to the uncrystallisable sugar in the molasses being optically active, and thus of themselves so act on polarised light as to vitiate the ordinary results; then direct polarisation cannot show the percentage of sucrose in a positive manner, it must therefore be estimated by optical inversion. This method depends on the fact discovered by Clerget, that when sucrose is inverted by hydrochloric acid the solution of invert sugar which results deviates the plane of polarised light to the left, and that of a normal solution showed  $100^\circ$  to the right, the same solution inverted would indicate  $44^\circ$  to the left at the temperature of  $0^\circ \text{C.} = 44 - \frac{T}{22}$

at the temperature  $T$ , so that the deviation would be nil at  $88^\circ \text{C.}$  Owing to other sugars, therefore, it is often necessary to make an inversion in the following way. The direct observation is made in the usual way, the remainder of the liquid is filtered, and a flask graduated at two points, 50 and 55 c.c., is filled to the 50 c.c. mark, and 5 c.c. concentrated fuming hydrochloric acid, sp. gr. 1.188 = 38 per cent. HCl, are added (that is, up to the 55 c.c. mark). The flask is shaken to make the liquid homogeneous, and inserted in a water bath kept at the temperature of  $68^\circ \text{C.}$  A thermometer is inserted in the flask, which is shaken so as to bring its contents uniformly to  $68^\circ \text{C.}$ , which takes about five minutes, after which the flask is kept at the same temperature of  $68^\circ$  to  $70^\circ \text{C.}$  for ten minutes longer, when the flask is withdrawn and cooled by immersion in water; when it has cooled down to  $20^\circ \text{C.}$  the thermometer is withdrawn, and 0.5 to 1 gramme of animal charcoal added, stirred and filtered rapidly. By the addition of the 5 c.c. of hydrochloric acid the volume of the sugar solution is increased by one-tenth; the optical properties of the latter are consequently weakened to the extent of one-tenth. In order to compensate for this weakness, a tube of 22 centimetres in length is taken instead of a 20-centimetre tube. Temperature almost without influence on sucrose rotation very perceptibly modifies that of invert sugar. The 22-centimetre tube therefore carries a thermometer to indicate the temperature of the sugar solution at the moment of the experiment. The liquid is transferred into a tube 22 centimetres in length, bearing a thermometer; the tube is then placed in the apparatus, and the operation conducted as before. The index of the compensator is no longer moved towards the left, but rather to the right; the number on the graduated scale is then read. This number is added to that obtained before inversion, and the richness in crystallisable sugar, which corresponds to this total at the temperature of the observation  $T$ , is then found by consulting *Clerget's* tables. If the index, after the action of the acid on the sugar solution, was still to the left of zero, it will be necessary to subtract these two numbers instead of adding them. In default of *Clerget's* tables, very ap-

proximate results may be obtained by means of the following formula :

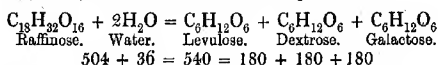
$$x = 1.635 \times \frac{200 S}{288 - T}$$

in which S represents the sum or difference of the numbers given by the instrument before and after inversion ;  $x$ , the number of grammes of sugar contained in one litre of the solution ; and, T, the temperature of the sugar solution. *Landolt* modified *Clerget's* formula thus :—

$$C = \frac{200 S}{284.8 - T}$$

in which C = crystallisable sugar ; S, the sum of the two saccharimetric readings before (P) and after ( $P_1$ ) the inversion, without taking the negative sign of the latter into account ; and T, the temperature of the last reading in °C. The value of C calculated in this way is compared with the result of the direct observation P. If C is greater than the direct observation, the sugar tested contains invert sugar. Otherwise there are dextro-rotary substances present.

*Raffinose or melitriose.*—Sugar refinery molasses contains a triose sugar—*raffinose* identical with *melitose* ( $C_{18}H_{32}O_{16}$ ), the sugar extracted from Australian manna. It may be separated from molasses by alcohol, in which it is less soluble than cane sugar in small crystals  $C_{18}H_{32}O_{16} + 5 \text{ Aq.}$  *Raffinose* is inverted into three glucoses, when its solution is heated with diluted sulphuric acid :—



*Estimation.*—A solution of *raffinose* is dextro-rotary ; it does not reduce *Fehling's* solution. Certain sugars, especially those extracted from molasses by the saccharate process, often contain *raffinose*, which, owing to its high dextro-rotary power (at 20° C.  $p = 0 - 10 + 104.5$  (constant)) being greater than that of crystallisable sugar, imparts an abnormal polarisation thereto. The total of the usual estimations therefore occasionally exceed 100 per cent., without taking into account undetermined substances. Moreover, even where the total sum of the percentages of the different estimation is less than 100, the excessive amount of the crystalline sugar is proved by the results of the optical inversion. But the formula given above does not suffice to rectify the percentage of crystallisable, and still less does it admit of the amount of *raffinose* being deduced from it. Both crystallisable sugar and *raffinose* may be easily ascertained by the use of *Creydt's* formula, deduced from direct experiments on mixtures of chemically pure samples of the two substances in known proportion. *Creydt* found that after a

TABLE LXXXI.—CLEROET'S TABLE.

10° C.	15° C.	20° C.	N.	N°.	10° C.	15° C.	20° C.	N.	N°.
1-39	1-37	1-34	1	1-64	63-99	62-82	61-66	46	75-22
2-78	2-73	2-68	2	3-27	65-38	64-19	63-00	47	76-85
4-16	4-10	4-02	3	4-91	66-77	65-56	64-34	48	78-48
5-56	5-46	5-36	4	6-54	68-17	66-92	65-68	49	80-12
6-95	6-83	6-70	5	8-17	69-57	68-29	67-03	50	81-75
8-35	8-19	8-04	6	9-81					
9-74	9-56	9-38	7	11-44	70-95	79-66	68-37	51	83-38
11-13	10-93	10-72	8	13-08	72-34	71-02	69-71	52	85-01
12-52	12-29	12-06	9	14-71	73-73	72-39	71-05	53	86-65
13-91	13-66	13-41	10	16-35	75-12	73-76	72-40	54	88-29
					76-51	75-12	73-74	55	89-93
15-30	15-03	14-75	11	17-99	77-90	76-49	75-08	56	91-56
16-69	16-40	16-09	12	19-62	79-29	77-85	76-42	57	93-20
18-08	17-77	17-43	13	21-26	80-68	79-22	77-76	58	94-83
19-47	19-14	18-77	14	22-89	82-07	80-59	79-10	59	96-46
20-86	20-51	20-11	15	24-52	83-46	81-94	80-43	60	98-10
22-26	21-88	21-45	16	26-16					
23-65	23-25	22-79	17	27-79	84-86	83-31	81-78	61	99-73
25-04	24-62	24-13	18	29-43	86-25	83-68	84-12	62	101-4
26-43	25-90	25-47	19	31-06	87-64	86-05	84-46	63	103-0
27-82	27-31	26-81	20	32-70	89-02	87-43	85-80	64	104-6
					90-41	88-80	87-14	65	106-3
29-21	28-68	28-15	21	34-34	91-81	90-16	88-48	66	107-9
30-60	30-05	29-49	22	35-98	93-20	91-54	89-82	67	109-5
31-99	31-42	30-83	23	37-61	94-59	92-90	91-16	68	111-2
33-38	32-79	32-16	24	39-25	96-00	94-25	92-50	69	112-8
34-77	34-16	33-51	25	40-88	97-38	95-60	93-83	70	114-4
36-17	35-53	34-85	26	42-51					
37-57	36-90	36-19	27	44-15	98-77	96-96	95-17	71	116-1
38-94	38-24	37-53	28	45-78	100-2	98-53	96-51	72	117-7
40-34	39-60	38-87	29	47-42	101-6	99-70	97-85	73	119-3
41-74	40-97	40-21	30	49-05	102-9	101-1	99-19	74	121-0
					104-3	102-4	100-5	75	122-6
43-12	42-38	41-55	31	50-69	105-7	103-8	101-9	76	124-2
44-51	43-70	42-89	32	52-33	107-1	105-2	103-2	77	125-9
45-90	45-07	44-23	33	53-97	108-5	106-5	104-5	78	127-5
47-20	46-43	45-57	34	55-60	109-9	107-9	105-9	79	129-1
48-68	47-80	46-91	35	57-24	111-3	109-3	107-2	80	130-8
50-08	49-16	48-25	36	58-87					
51-47	50-53	49-59	37	60-50	112-7	110-9	108-6	81	132-4
52-86	51-90	50-93	38	62-14	114-1	112-0	109-9	82	134-1
54-25	53-26	52-27	39	63-77	115-5	113-3	111-3	83	135-7
55-64	54-63	53-63	40	65-40	116-9	114-7	112-6	84	137-3
					118-2	116-1	113-9	85	139-0
57-03	55-99	54-96	41	67-08	119-6	117-4	115-3	86	140-6
58-42	57-36	56-30	42	68-67	121-0	118-8	116-6	87	142-2
59-81	58-73	57-64	43	70-31	122-4	120-2	118-0	88	143-9
61-20	60-09	58-98	44	71-95	123-8	121-5	119-3	89	145-5
62-59	61-46	60-32	45	73-58	125-2	122-9	120-6	90	147-1

TABLE LXXXI.—continued.

10° C.	15° C.	20° C.	N.	N'.	10° C.	15° C.	20° C.	N.	N'.
126.6	124.8	122.0	91	148.7	154.4	151.6	148.8	111	181.5
128.0	125.6	123.8	92	150.4	155.8	153.0	150.1	112	183.1
129.4	127.0	124.7	93	152.1	157.2	154.4	151.5	113	184.7
130.8	128.4	126.0	94	153.7	158.6	155.7	152.8	114	186.4
132.2	129.7	127.4	95	155.3	160.0	157.0	154.2	115	188.0
133.6	131.1	128.7	96	156.9	161.3	158.4	155.4	116	189.7
134.9	132.5	130.0	97	158.6	162.7	159.8	156.8	117	191.3
136.3	133.8	131.4	98	160.2	164.1	161.2	158.2	118	192.9
137.7	135.2	132.7	99	161.9	165.5	162.5	159.5	119	194.6
139.1	136.6	134.0	100	163.5	166.0	163.9	160.8	120	196.2
140.5	137.9	135.4	101	165.1	168.3	165.3	162.2	121	197.8
141.9	139.3	136.7	102	166.8	169.7	166.6	163.5	122	199.5
143.3	140.7	138.1	103	168.4	171.1	168.0	164.9	123	201.1
144.7	142.0	139.4	104	170.0	172.5	169.4	166.2	124	202.7
146.0	143.4	140.8	105	171.7	173.9	170.7	167.6	125	204.4
147.4	144.8	142.1	106	173.3	175.3	172.1	168.9	126	206.0
148.8	146.1	143.4	107	174.9	176.6	173.5	170.2	127	207.6
150.2	147.5	144.8	108	176.6	178.0	174.8	171.6	128	209.3
151.6	148.8	146.1	109	178.2	179.4	176.2	172.0	129	210.9
153.0	150.2	147.4	110	179.8	180.8	177.5	174.2	130	212.6

solution of cane sugar indicating  $+100^\circ$  of the saccharimeter had been inverted, as previously described, the inverted solution at a temperature of  $20^\circ$  C. showed a deviation of  $-32^\circ$ . But a solution of raffinose, showing  $+100^\circ$  by the saccharimeter, gives after inversion a deviation to the right of  $50^\circ.7$  at  $20^\circ$  C. A mixture of the two substances, in definite proportions, similarly treated should yield an intermediate rotation. Let A = Polarisation before inversion, B = Polarisation after inversion at  $20^\circ$  C., C = Difference between A and B, the amount the polarisation has been reduced: the percentage of crystallisable sugar S, and of raffinose R, may then be calculated from the following formulæ:—

$$(a) S = \frac{C - 0.493 A}{0.827} = 0.613 A - 1209 B$$

$$R = 1.017 A - \frac{C}{21.98}.$$

This calculation is only applicable to those cases where only these two optically active substances are present. With more complicated mixtures, the result is less exact the greater the amount of other substances present. A German authority gives the following formulæ:—

$$R = \frac{I + 51.24 \times P}{0.8390}$$

in which P = polarisation before inversion, and I = polarisation after inversion.

*Estimation of raffinose in sugar products.*—(a) *First products.*—100 grammes are shaken in a stoppered, narrow-necked 250 c.c. flask with 150 c.c. of commercial wood spirit, previously mixed with a few drops of potash alum solution to neutralise the alkalinity of the sugar. The precipitated sulphates also carry down colouring matter. A 100 c.c. flask is filled to the mark with the solution, 40 c.c. removed by distillation, 20 c.c. of water added, and lead acetate as long as a precipitate forms. A little moist aluminium hydrate is then added, the flask filled to the mark with water, 5 c.c. of hydrochloric acid (36 per cent. HCl) added, and heated for ten minutes at 68° C., cooled rapidly to 20° C., and polarised in the 200-millimetre tube. The value of raffinose calculated from the equation and multiplied by 1.5 = the percentage of raffinose within 0.05 per cent. of raffinose, may be estimated in this way, but to extract such small amounts some potassium acetate must be added to the solvent. (b) *For solid after-products.*—As the raffinose in these is only dissolved very slowly and incompletely by methyl alcohol, the process has to be modified. 30 grains of raw sugar are treated in a graduated flask with 6 to 9 c.c. of water and enough potash alum solution to neutralise the alkalinity of the sugar. The sugar is then dissolved by heat, and about 120 c.c. of wood spirit added, with continual shaking. After cooling, it is made up to the mark with wood spirit, a little powdered sugar added, and the flask shaken frequently during one hour. After from 10 to 11 c.c. of wood spirit have been added to correct for the volume of sugar separated, the liquid is filtered and the estimation continued as directed under (a). The value for raffinose found, multiplied by 5, gives the percentage of raffinose. These estimations are accurate within 0.1 per cent. of raffinose. (c) *Molasses.*—On account of solubility of molasses in methyl alcohol or wood spirit no concentration of the raffinose can take place, and the method is therefore very similar to the German inversion method. 12 grammes molasses are mixed in a 100 c.c. flask with 12 grammes water, and the amount of potash alum solution necessary for neutralisation dissolved, and wood spirit added to the mark. It is then shaken, filtered, and filtrate analysed as under (a). The value found, multiplied by 12.5, gives the percentage of raffinose present. In America the estimation of raffinose in raw sugars is made by a method only slightly differing from the German one. The Austro-Hungarian Centralverein had several imported and a high-grade home-produced table syrup examined by Herzfeld's methods. The results were as follows :—

TABLE LXXXII.—TABLE SYRUPS—ANALYTICAL DATA.

	Imported Table Syrups.				Domestic Syrup.
	I.	II.	III.	IV.	V.
Specific gravity at 17°·5 C. .	1·4283	1·4178	1·4344	1·4309	1·4181
Degrees Balling . . .	81·10	80·22	82·74	82·23	80·33
Direct polarisation . . .	38·50	37·80	36·05	32·90	52·50
Polarisation at 20° C. after inversion . . .	16·1	17·0	16·50	16·0	17·0
Reducing sugar determined as invert sugar according to Preuss . . .	25·50	21·56	28·34	21·67	6·47
Total solids . . .	74·18	71·04	74·03	74·28	77·55
Ash . . .	3·75	5·07	4·68	6·94	9·74

The following composition is calculated for the syrups from these analyses :—

TABLE LXXXIII.—TABLE SYRUPS—COMPOSITION.<sup>1</sup>

	I.	II.	III.	IV.	V.
Water . . . . .	23·37	28·96	25·97	25·72	22·45
Cane sugar . . . . .	41·91	41·31	39·60	37·30	52·45
Invert sugar . . . . .	7·09	10·32	10·44	12·94	6·47
Optically inactive sugar . . .	18·41	11·24	17·90	8·73	0·00
Ash . . . . .	5·75	5·07	4·68	6·94	9·74
Organic matter not sugar, by difference . . . . .	2·97	3·10	1·41	8·37	8·89
	100·00	100·00	100·00	100·00	100·00

Optically inactive sugar is here a mixture of glucose and invert sugar, in which the right-handed rotation of the one is exactly equal to the left-handed rotation of the other. These analyses show that the domestic syrup is nothing more than a beet-sugar molasses, whilst the imported syrups are a better grade of cane molasses. The taste and general appearance of the imported syrups showed them to be cane molasses, whilst the home table syrup did not taste much better than common molasses.

<sup>1</sup> See Table LXXX., p. 396, and Table LXXXIV., p. 404.



TABLE LXXXIV.—ANALYSES OF MOLASSES FROM VARIOUS SOURCES.

	Java Prinsen Geerligs.			Thompson Average from Different Sources.
	Extreme Variations.		Average.	
	From	To		
Crystallisable sugar calculated by in- version . . . . .	5.7	44.3	35.3	26.34
Reducers . . . . .	18.8	39.4	27.6	28.18
Dextrose . . . . .	8.2	22.9	14.2	...
Levulose . . . . .	5.6	16.5	13.4	...
Ash (Total) . . . . .	4.53	9.04	8.08	8.26
Water . . . . .	17.1	42.3	19.1	26.6
Organic matter . . . . .	...	...	11.23	10.67
Density . . . . .	1.315	1.481	1.481	...
Acidity as acetic acid . . . . .	...	0.5	0.19	...
Real purity in ratio to sucrose . . . . .	9.8	53.7	43.6	...
Nitrogenous matter . . . . .	...	...	...	2.21

## SCHEME A.—OFFICIAL GERMAN METHODS USED IN TAXING SUGARS.

DIRECTIONS FOR THE EXAMINATION OF SYRUPS WHICH CONTAIN 2 PER CENT. OR MORE OF INVERT SUGAR, STARCH SUGAR OR RAFFINOSE, AS ALSO OF SUGARS CONTAINING RAFFINOSE.

In examination of syrups handed over to the chemist because they contain 2 per cent. or more of invert sugar, the determination of specific gravity and degree Brix can be made as directed in Article A. The direct determination of specific gravity by the pycnometer can take the place of this method, but it is not allowable to substitute the estimation of total solids, as this would (1) cause differences in coefficient of purity as obtained by officers and chemists, and (2) the determination of total solids in syrups containing invert sugar is far too difficult and takes too much time to be of any use in everyday practice. In calculating coefficient of purity the method used in the sugar-house, *viz.*, considering only sucrose as sugar, cannot be used, but the invert sugar must be calculated as sucrose by deducting one-twentieth of the amount found, adding to the direct content of sucrose and basing the calculation on this result. For determination of the sugar-content various methods can be applied according to amount of invert sugar, starch sugar or raffinose present.

The invert sugar of syrups is very often inactive, but may be levo-rotary, which deviation, according to late investigations, is 0.33, according to others, 0.34 times as great as the dextro-rotation of

sucrose. As soon, therefore, as considerable invert sugar is present, the polarisation of the sucrose present will be diminished *pro rata*. Meissl, in the case of colonial sugars, multiplied the invert sugar by 0.34, added this to the polarisation, and thus arrived at the correct amount of sucrose. This method would, however, be out of place in the analysis of syrups, as the invert sugar present very often does not show its normal rotatory power, but varies down to complete inactivity. Meissl's correction would then lead to unreliable results. At the same time the rotatory power of the invert sugar may cause the results to be too low. It therefore becomes obvious that the determination of total sugar from the polarisation and the invert sugar found is only possible when the invert sugar present does not exceed a certain percentage; e.g., if 6 per cent. of invert sugar were present the amount of sucrose could be  $6 \times 0.33 = 1.98$  per cent. too low. Therefore, as syrups handed to the chemist for examination may contain considerable quantities of invert sugar whose rotatory power we do not know, we recommend the rejection of the optical method completely, and the adoption of a gravimetric one, a new and easy modification of which will be given under I. The presence of starch sugar or raffinose makes a difference. The reducing power of starch sugar, which in the commercial article presents a variation corresponding to from 40 to 60 per cent. of sugar, is not changed by the conditions under which the inversion of sugar syrups is made in the gravimetric method; and as we cannot determine the quantity of starch sugar present accurately, the gravimetric method for estimation of total content of beet sugar and of quotient of purity is no longer applicable. It would lead to large errors, and syrups having a coefficient of purity of over 70, would, if examined by this method, after the addition of a certain quantity of starch sugar, appear to be below 70. In such cases, where starch sugar is present the influence exerted by the levo-rotation of the invert sugar on the polarisation can no more be considered, as starch sugar has a higher dextro-rotatory power than the other sugars present. To avoid mistakes which could be easily caused by adding starch sugars to syrups of a coefficient of purity of over 70, it is best when starch sugar is present to calculate the real sucrose-content from the polarisation, and the invert sugar directly determined. This method is described under II. If raffinose is present still another method given in III. must be adopted.

I.—The presence of starch sugar need not be considered. The examination of syrups which do not contain any starch sugar syrup will be a common occurrence, as such an addition is generally not made by the manufacturers, but by the dealers. One operation will here suffice for the determination of total sugar: Weigh out half the normal weight, 13.024 grammes of syrup, dissolve in a 100 c.c. flask in 75 c.c. of water. Add 5 c.c. HCl (38.8 per cent. HCl) and warm

in a bath to  $67^{\circ}$  to  $70^{\circ}$  C. As soon as the contents of the flask have reached this temperature, heat for five minutes, not allowing the temperature to rise above  $70^{\circ}$  C., and shaking frequently. As the previous warming will take two and a half to five minutes, the whole operation will require from seven and a half to ten minutes. Fill up to mark, shake, and dilute 50 c.c. of this solution to a litre. Transfer by a pipette 25 c.c. of this to a flask, and add 25 c.c. of a solution of sodium carbonate (1.70 grammes anhydrous salt to the litre) to neutralise free acid. Add 50 c.c. Soxhlet's solution, heat to boiling as in the determination of moist sugar, and keep the liquid in ebullition for three minutes. As all the sucrose in this solution has been inverted, and is therefore unable to influence the result, we need not be so careful about the time of boiling. Soxhlet's experiments show that two or three minutes' difference will not influence result materially. Dilute the liquid with an equal volume of water, which has been previously boiled so as to expel the air, and continue as direct under determination of invert sugar. The tables so far published for the calculation of results are of no value in this case, as they have been constructed with regard to glucose, or a mixture of invert sugar and saccharose. The following table (p. 407) has been compiled for invert sugar, and enables the analyst to calculate the amount of sucrose directly from the amount of copper found.

*Example.*—25 c.c. of the solution = 0.7628 gramme substance gave 0.1628 gramme of copper, which corresponds to 0.082 gramme of sugar. Therefore, the syrup contained 50.4 per cent. sugar.

Supposing that this syrup showed  $80^{\circ}$  Brix, then its coefficient would be 63. This coefficient is only calculated to tenths, hundredths in the case of the degree Brix being taken as an additional tenth, while with the coefficient they are not considered; thus  $82.85^{\circ}$  Brix would be read  $82.9$ , while a coefficient of  $69.99$  is taken as  $69.9$  and not  $70$ .

II.—The syrup to be examined may contain starch sugar.

In this case a direct polarisation must first be made. If this gives a coefficient of purity above  $70$ , further examination is unnecessary, as it would only tend to raise the coefficient, never to lower it.

If, however, the coefficient falls below  $70$ , the presence of starch sugar is still possible. To determine whether any is present the syrup is inverted as in I., and made up to the mark.

It is then decolorised by adding  $\frac{1}{2}$  to  $1$ , in the case of dark syrups from  $2$  to  $3$  grammes of bone charcoal, previously washed with hydrochloric acid, or of charred blood. If the latter is used, its absorption factor for invert sugar must be previously determined, and a corresponding correction in the polarimetric reading made in case the left-handed rotation is to be estimated accurately. In the present case it is sufficient to approximately determine this rotation at about  $20^{\circ}$ . Experience has shown that unadulterated syrups do

not always have a levo-rotation corresponding to 0.33 of the original right-handed rotation, but in all cases it is at least one-fifth of the original reading. A syrup polarising 55 directly should, after inversion, read at least -11, calculated for normal weight. If this syrup should read -10 or less, or should even rotate to the right, then it has

TABLE LXXXV.—TABLE FOR CALCULATING THE SUCROSE CORRESPONDING TO THE INVERT SUGAR PRESENT FROM THE COPPER FOUND ON THREE MINUTES' BOILING.

Sucrose. Mg.	Copper. Mg.	Sucrose. Mg.	Copper. Mg.	Sucrose. Mg.	Copper. Mg.	Sucrose. Mg.	Copper. Mg.
40	79.0	73	145.2	106	208.6	139	269.1
41	81.0	74	147.1	107	210.5	140	270.9
42	83.0	75	149.1	108	212.3	141	272.7
43	85.2	76	151.0	109	214.2	142	274.5
44	87.2	77	153.0	110	216.1	143	276.3
45	89.2	78	155.0	111	217.9	144	278.1
46	91.2	79	156.9	112	219.8	145	279.9
47	93.3	80	158.9	113	221.6	146	281.6
48	95.3	81	160.8	114	223.5	147	283.4
49	97.3	82	162.8	115	225.3	148	285.2
50	99.8	83	164.7	116	227.2	149	286.9
51	101.3	84	166.6	117	229.0	150	288.8
52	103.3	85	168.6	118	230.9	151	290.5
53	105.8	86	170.5	119	232.8	152	292.3
54	107.3	87	172.4	120	234.6	153	294.0
55	109.4	88	174.3	121	236.4	154	295.7
56	111.4	89	176.3	122	238.3	155	297.5
57	113.4	90	178.2	123	240.2	156	299.2
58	115.4	91	180.1	124	242.0	157	300.9
59	117.4	92	182.0	125	243.9	158	302.6
60	119.5	93	183.9	126	245.7	159	304.4
61	121.5	94	185.8	127	247.5	160	306.1
62	123.5	95	187.8	128	249.3	161	307.8
63	125.4	96	189.7	129	251.2	162	309.5
64	127.4	97	191.6	130	252.9	163	311.3
65	129.4	98	193.5	131	254.7	164	313.0
66	131.4	99	195.4	132	256.5	165	314.7
67	133.4	100	197.3	133	258.3	166	316.4
68	135.3	101	199.2	134	260.1	167	318.1
69	137.3	102	201.1	135	261.9	168	319.9
70	139.3	103	202.9	136	263.7	169	321.6
71	141.3	104	204.8	137	265.5	170	323.3
72	143.2	105	206.7	138	267.3		

been adulterated with starch sugar. When the absence of starch sugar has thus been proved the syrup must be examined according to I.

If starch sugar be present then we must arrive at the total sucrose by adding to the direct polarisation the invert sugar found by Fehling's solution. In this procedure Fehling's solution is used according to Soxhlet's directions, but as the amount of sugar taken by this

author would not suffice for 10 grammes of substance, we have to make preliminary experiments to determine the quantity of syrup which should be taken. This is done by diluting 10 grammes syrup to 100 c.c., placing 5 c.c. of Fehling's solution into several test-tubes and adding successively 8, 6, 4 and 2 c.c. of the syrup solution. On boiling we will arrive at a point where the Fehling's solution is no longer decolorised. If this is the case with 6 c.c. then 6 grammes of substance are weighed out, dissolved in 50 c.c. of water and 50 c.c. Fehling's solution added. The whole is boiled for two minutes, and the analysis conducted as in the estimation of invert sugar. The invert sugar is calculated according to Meissl's table:—

Let I.  $\frac{Cu}{2}$  = approximate amount invert sugar = Z.

II.  $Z \times \frac{100}{p}$  = approximate per cent. invert sugar = y.

III.  $\frac{100 Pol}{Pol + y} = R$ , relative number for sucrose.

$100 - R = I$  relative number for invert sugar.

R : Z relation of sucrose to invert sugar = 6.

IV.  $\frac{Cu}{p} \times F$  = correct percentage of invert sugar.

In these formulæ Cu is the weighed copper; p the amount of substance taken; Pol the polarisation; Z facilitates reading the vertical column, and R : Z the horizontal column of the following table.

To use the table find the columns agreeing most closely with the values found for Z and R : Z. When these columns meet the value of factor F will be found.

TABLE LXXXVI.—DETERMINATION OF OVER 1.5 PER CENT. GLUCOSE IN SUGAR (MEISSL'S).

Sucrose : Invert Sugar = R : Z	Milligrammes Invert Sugar = Z.								
	245	225	200	175	150	125	100	75	50
90 : 10	F.	F.	F.	F.	F.	F.	F.	F.	F.
91 : 9	56.2	55.1	54.1	53.6	53.1	52.6	52.1	51.6	51.2
92 : 8	56.2	55.1	54.1	53.6	53.1	52.6	52.1	51.6	51.2
93 : 7	56.2	55.1	54.1	53.6	53.1	52.6	52.1	51.6	51.2
94 : 6	55.7	54.1	53.1	52.6	51.6	50.7	50.3	49.8	49.8
95 : 5	55.7	53.6	52.6	52.1	51.2	50.3	49.4	48.9	48.5
96 : 4	...	...	52.1	51.2	50.7	49.8	48.9	47.7	46.9
97 : 3	...	...	50.7	50.3	49.8	48.9	47.7	46.2	45.1
98 : 2	...	...	49.9	48.9	48.5	47.3	45.8	43.3	40.0
99 : 1	...	...	47.7	47.3	46.5	45.1	43.3	41.2	38.1

F = Factors.

*Example.*—The polarisation of a sugar is 86.4, and 3.256 grammes substance =  $p$  is equivalent to 6.290 grammes copper =  $Cu$ , then

$$I. \frac{Cu}{2} = \frac{0.290}{2} = 0.145 = Z.$$

$$II. Z \times \frac{100}{p} = 0.145 \times \frac{100}{3.256} = 4.45 = y.$$

$$III. \frac{100 \times Pol}{Pol + y} = \frac{86.40}{86.4 + 4.45} = 95.1 = R.$$

$$100 - R = 100 - 95.1 = Z : R : Z = 95.1 : 4.9.$$

By consulting the table we find that column 150 comes nearest to  $Z = 145$  and column 95 : 5 is nearest to  $R : Z = 95.1 : 4.9$ . Where these columns meet we find the factor 51.2 which enters into the last calculation.

$$IV. \frac{Cu}{p} \times F = \frac{0.290}{3.256} \times 51.2 = 4.56 \text{ per cent, invert sugar.}$$

To convert this into saccharose we subtract  $\frac{1}{50}$ ;  $4.56 - 0.23 = 4.33$ ; add this figure to the direct polarisation and calculate the co-efficient of purity from this and the degree Brix.

III.—*The presence of raffinose must be considered.*—(a) The direct polarisation of the sugar is determined as also (b) the indirect polarisation at exactly 20° C. The same precautions must be taken in making the inversion as in I. and II. Half the normal weight is dissolved in 75 c.c. of water in a 100 c.c. flask, 5 c.c. 38.8 per cent. HCl added, heated for  $7\frac{1}{2}$  to 10 minutes to 60° to 70° C. After making up to mark and clarifying with washed bone black the reading is taken at exactly 20° C. The following formula serves to calculate the result:—

$$Z(\text{sugar}) = \frac{0.5188 P - J}{0.845} \text{ and } R(\text{raffinose}) = \frac{P - Z}{1.85} \text{ when } P = \text{direct}$$

polarisation and  $J$  invert reading for normal weight, the prefixed sign having been changed. The invert sugar is determined as in other syrups. If test given in A shows it to be present in quantities of less than 2 per cent., then it need not be further considered. Two per cent. or more of invert sugar must be determined quantitatively according to Meissl's method described in II. and calculated as saccharose. In finding the factor of this table raffinose is to be considered as sucrose and Meissl's value.  $Pol$  is therefore the sum of sucrose and raffinose. The coefficient of purity is calculated from the degree Brix and the total sucrose (that is sucrose + invert sugar calculated to sucrose), the raffinose not being considered.

*Example.*—The examination of a syrup gave 85.6 Brix, 76.6 direct polarisation - 3  $Pol$  after inversion. By using above formula we get 50.5 sucrose and 14 raffinose. There were also found 2.1 per cent. sucrose as invert sugar. Therefore the total

sucrose is 52.6 and the coefficient 61.4. Rank fraud might be practised by adding small amounts of starch sugar to very pure syrups and requesting an examination in regard to raffinose present. In such cases above method would cause an error in finding too small a sugar-content and a considerable amount of raffinose corresponding to starch sugar added. If starch sugar is known to be present, then the method described in II. must be used. The estimation of starch sugar cannot be made exactly as described in II. for syrups in general, as syrups containing raffinose give a different invert reading. If therefore a product is to be examined which is supposed to contain raffinose, it is best to use method III. and calculate sucrose and raffinose from that. The observed readings should not vary more than 5° from the calculated readings, otherwise starch sugar is certainly present and the raffinose formula no more applicable. In such a case the syrup must be examined according to II.

*Examination of sugars for raffinose.—The official German method.*—The method of examining syrups containing raffinose can be applied to sugars. Direct polarisation is taken as usual, indirect after inversion of one-half normal weight as directed for syrups, p. 409, and the sucrose and raffinose contents calculated by means of the formula given. Numerous experiments show this method gives reliable results. Thus there were found in mixtures of sucrose and raffinose:—

TABLE LXXXVII.—DETERMINATION OF RAFFINOSE IN RAW SUGAR.

Mixture.		Found by the Method.	
Sucrose.	Raffinose.	Sucrose.	Raffinose.
Per Cent.	Per Cent.	Per Cent.	Per Cent.
97.00	8.00	97.02	2.98
91.00	9.00	90.99	8.95
85.00	15.00	85.06	14.97

Though this method is accurate, a wide limit must be given for experimental errors. A variation of 0.6 between sucrose as calculated by the raffinose formula and that determined directly by polarisation has therefore been made the limit. Suppose, e.g., that the sucrose found by direct polarisation is 92.6 and that calculated by the formula 93 per cent., then this difference is due to errors in method. Raffinose should then be reported absent and the direct polarisation used for sugar-content. But if only 91.9 per cent. sucrose were found by the formula while the direct polarisation was 92.6, then raffinose is undoubtedly present. To avoid mistakes caused by larger errors than 0.6, it is advisable to make a control determination

where the difference is less than 1 per cent., and, to base presence or absence of raffinose upon result of this control. The above limit for errors will not influence utility of method, as percentage of raffinose observed in high sugars always corresponds to more than 0.6 per cent. of sucrose. No method has yet been proposed for determination of smaller amounts of raffinose—they may be neglected. Scheibler's method of calculating small amounts of raffinose by supposing organic substances not sugar to be equal to ash is not accurate, as excess of organic substance over ash is not known. The method is of value in cases where result found by the formula varies comparatively little from the polarisation; *e.g.*, corresponding to less than 1 per cent. of sucrose. Here it will decide whether raffinose is present or whether the difference is caused by errors. Polarisation, moisture and ash are determined, the organic substances not sugar taken as equal to the ash, and these four constituents added together. Where raffinose is present the result will be over 100; if it falls below 100, raffinose may be considered absent. In the former case, the percentage of raffinose is calculated as follows: The percentage of water plus twice the ash is deducted from 100. The result corresponds to the percentage of sucrose plus anhydrous raffinose. If we substitute  $a$  for the figure thus found,  $p$  for the direct polarisation,  $x$  for the sucrose present, and  $y$  for the raffinose present, then

$$\begin{aligned}x + 1.85 y &= p \\x + y &= a \\x (\% \text{ sucrose}) &= \frac{1.85 a - p}{0.85} \\y (\% \text{ raffinose}) &= \frac{a - 1.85 a - p}{0.85}\end{aligned}$$

The limit for error must here be placed at 0.3; that is, the sum of polarisation, twice the ash and moisture, must be more than 100.3 if the method is employed. Otherwise the method would be too uncertain for practice, as errors of 0.2 may easily occur in the direct reading. The following example has been so constructed as to show that the method with the above limit of error is not capable of determining variations of 0.6 per cent.

A sugar gave 99.7 polarisation, 0.4 water and 0.1 ash, then the sum total was

99.7	$a = 100.0$
+ 0.4	- 0.4
+ $2 \times 0.1 = 0.2$	- 0.2
100.3	$a = 99.4 \quad p = 99.7$

Therefore  $x$  sucrose = 99.05 which is taken as 99.1, and  $y$  raffinose = 0.3. It is apparent that the 0.3 plus polarisation found by this method, and which must be taken as the limit of error, corresponds



exactly to the variation between actual sucrose and direct reading, which has been made the limit for the inversion method with raffinose formula. The control is unnecessary when a difference of 1 per cent. or more is found between the actual sucrose and direct reading. Very often the result of the raffinose formula is taken even if the control gives a negative result. If Scheibler's method gives negative results with a sugar varying less than 1 per cent., then raffinose should be reported absent. If, however, the control leaves any doubt as when the sum varies between 100 and 100.3, or if raffinose be actually found, then the results obtained by the inversion method should always be reported so long as the difference between the sucrose found and the direct readings amount to more than 0.6 per cent. If it is less than 0.6 or just 0.6 then the analyst should report that he is unable to detect raffinose. In calculating the final result hundredths should be taken as an additional tenth, thus 97.01 per cent. sucrose is 97.1.

SCHEME B.—GERMAN OFFICIAL INSTRUCTIONS FOR THE EXAMINATION OF CHOCOLATE, CANDIES AND LIQUORS FOR THE QUANTITY OF SUCROSE CONTAINED THEREIN.

In all cases where the substance to be examined contains no other optically active bodies, the direct polarisation will give accurate results. If such are present, as for instance glucose, invert sugar, maltose, dextrine, pectinous bodies, the application of the polarisation becomes uncertain, and only in a few cases given below can somewhat reliable results be obtained. As regards the preparation of the solutions the following must be remembered: Material which consists mostly of sucrose and leaves only a small residue on being treated with water, can be weighed out in the dish, and the solution immediately transferred to the sugar flask. When the substance, however, contains much insoluble matter, the latter should not be washed into the flask, as the volume of the sugar solution in such a case would be less than 100 c.c. In this case the residue must be separated from the solution by filtration and washing. Generally the sugar solution is not quite transparent and must be treated with some clearing agent. The solutions serving this purpose are: (1) Lead acetate, of which from 1 to 10 c.c. are mixed with the solution and the latter filtered after standing from a quarter to half an hour. (2) Lead acetate with subsequent addition of alum or aluminium sulphate, the lead sulphate formed carrying down the foreign matter. (3) Aluminium hydrate in the form of a thin paste, of which several cubic centimetres are shaken with the solution. (4) Tannic acid to precipitate albuminous substances. This must be examined beforehand with regard to its optical activity. (5) Charred blood or bone black to remove colouring matter,  $\frac{1}{2}$  to 1 gramme, are added

to the liquid. In some cases this clearing is difficult, and the method best adapted must be determined by previous experiments. For solutions prepared from candies the pasty aluminium hydrate is the best medium. When, as is often the case, invert sugar is present, the result of the direct polarisation will be too low on account of the left-handed rotation of this sugar. In such a case Clerget's inversion method must be applied to find the correct per cent. of sucrose. This is conducted as follows: 26.048 grammes are dissolved in a sugar flask and the solution made up to 100 c.c.; 50 c.c. of this solution are transferred by means of a pipette to a 50 or 55 c.c. flask, cleared and polarised, the reading being corrected for the extra 5 c.c. The liquid adhering to the pipette is washed into the 100 c.c. flask containing the remaining 50 c.c. (13.024 grammes), 5 c.c. of concentrated hydrochloric acid (38 per cent., sp. gr. 1.18 at 15° C.) added, and the flask heated for fifteen minutes at 67° to 70° C. in a water bath. The temperature should not exceed this limit. The flask is then cooled rapidly and the solution made up to 100 c.c. If the liquid is coloured, it is shaken with  $\frac{1}{2}$  to 1 gramme of bone black and filtered through a double filter. It is then polarised in a 200 mm. tube which is provided with a thermometer. As the rotatory power of invert sugar is much influenced by the temperature, this factor must be regarded. The reading is best taken between 18° and 22° C. and the temperature accurately determined. The reading must also be doubled on account of the dilution of the liquid. To calculate per cent. of sucrose (R) the two readings are added together, the sum (S) multiplied by 100 and divided by  $142.4 - \frac{1}{2}t$  where  $t$  is the temperature at which the invert reading was taken. If the temperature is exactly 20° C., the result can be made more accurate by substituting 142.66 for 142.4, thus:

$$R = \frac{100 S}{142.66 - \frac{20}{2}} = \frac{100 S}{132.66} = 0.7538 S.$$

If very much invert sugar is present, both the direct and indirect readings must be taken at the same temperature.

I. *Chocolate*.—Weigh out in a German-silver dish 13.024 grammes of the rasped chocolate, moisten with alcohol (to aid the solution in water), add 30 c.c. of water, and heat for ten to fifteen minutes on the water bath. Filter while warm into 100 to 110 c.c. flask, using a folded filter, any turbidity of the filtrate not being of any consequence. The residue on the filter is washed with hot water until the filtrate amounts to about 100 c.c. Five c.c. of lead acetate are then added, the liquid allowed to stand fifteen minutes, and a few drops of alum solution and some moist aluminium hydrate added. The liquid is then made up to 110 c.c., shaken well, and filtered through a folded filter. Slight moistening of the filter will aid filtration, but the first 25 c.c. of filtrate should

then be rejected. The polarisation should be increased by one-tenth and then doubled.

II. *Candies and confectionery*, (a) *Dragees* (seeds or kernels coated with sugar and flour).—26·048 grammes are covered in a beaker with 40 to 50 c.c. of water and allowed to stand, stirring now and then, until the mass is completely saturated. If the liquid shows an acid reaction, some precipitated calcium carbonate or a few drops of ammonia are added. The larger particles are now separated by filtering through cloth, the filtrate being collected in a 100 to 110 c.c. flask. The residue on the filter is washed with cold water until about 100 c.c. of filtrate have accumulated. Some aluminium hydrate is then added to clear it, the solution made up to 110 c.c. with water, and about half a gramme of bone black added in case the liquid is coloured. It is then allowed to stand half an hour, with occasional shaking. The solution is finally filtered through a dry folded filter.

A preliminary test for invert sugar is made with copper sulphate and soda lye, and the solution analysed accordingly. Dragees almost always contain invert sugar.

(b) *Raffinade wafers* (cane sugar, with ethereal oils and colouring matter).—26·048 grammes dissolved in water, made up to 100 c.c. in flask, and, if necessary, decolorised with bone black. A preliminary test for invert sugar must always be made and the analysis directed accordingly.

(c) *Santonin wafers* (worm wafers, cane sugar, with santonin and some agglutinant, as egg albumen).—13·024 grammes dissolved in a 100 c.c. flask (the santonin being insoluble), 5 c.c. of lead acetate added, with a few drops of alum solution, the solution allowed to stand some time, with occasional shaking, made up to 100 c.c. and filtered.

(d) *Dessert bonbons* (Fondant's, Pralinees, chocolate bonbons, consisting of cane sugar or invert sugar, serving as a covering for marmalade, fruit or chocolate).—13·024 grammes are treated with water and a few drops of ammonium hydrate until dissolved. If only a slight residue remains the whole can be immediately transferred to a 100 c.c. flask, otherwise it must first be filtered. One-half of the liquid is inverted, the other polarised directly after having been cleared by means of aluminium hydrate in a 50 to 55 c.c. flask.

(e) *Marzipan Masse* (cane sugar, with crushed almonds).—13·024 grammes material are triturated in a porcelain mortar with cold water, then mixed in a flask with 50 c.c. of water and about 30 c.c. of pasty aluminium hydrate, well shaken and filtered into a 200 c.c. flask. The residue on the filter is washed with water until the filtrate amounts to 200 c.c. As this candy contains no invert sugar the solution can be polarised directly in the 200 mm. tube, the reading being multiplied by 4 to correct for the dilution.

(f) *Cakes and sweetened pastry*.—26·048 grammes of the powdered

material are mixed in a flask with about 75 c.c. alcohol (85 to 90 per cent.) and allowed to stand one-half hour in a warm place. It is then filtered through a fine cloth filter and the residue washed several times with alcohol. The filtrate is collected in a porcelain dish and heated on the water bath until the alcohol has all been driven off. One-half gramme bone black is then added and the solution filtered into a 100 c.c. flask. 50 c.c. are used for inversion, the remainder polarised directly.

(g) *Sugar-coated and preserved fruits* (marmalade, compots, jellies).

—A considerable amount of invert sugar and pectinous bodies are always present. The aqueous solution of the latter has no rotatory power. If the material is solid in consistence, a crushed or finely cut average sample must be prepared; 13.024 grammes are then mixed with 80 to 50 c.c. water and a few drops of ammonium hydrate (to neutralise any free vegetable acids), and allowed to stand several hours. It is then passed through a cloth filter into a 100 or 200 c.c. flask, the residue washed several times with hot water and about 10 c.c. of aluminium hydrate and one-half gramme bone black added to the filtrate. It is then shaken up and made up to the mark. After filtering the filtrate is polarised according to Clerget's method. Fruit, jellies and marmalade are analysed in a similar manner. If the sucrose articles enumerated under II. (g) were calculated by the

formula  $R = \frac{100 S}{42.4 - \frac{1}{2} t}$ , we would only obtain the percentage present at the time of analysis. The acids of the fruit have, however, converted a large amount of the cane sugar originally used into invert sugar. The original percentage of cane sugar, which is taken as a basis for taxation by the custom house, can be calculated from the reading of the inverted solution. If this reading is referred to 26.048 grammes material dissolved in 100 c.c. and polarised in a 200 mm. tube, we have the following relations, if the above figure is called B: A solution of 26.048 grammes of cane sugar in 100 c.c. has after inversion at the temperature  $t$  the left-handed rotation  $42.4 - \frac{1}{2} t$ . The amount of cane sugar corresponding to the observed polarisation B can thus be calculated by the proportion—

$$42.4 - \frac{1}{2} t : 26.048 = B : \frac{26.048}{42.4 - \frac{1}{2} t}$$

and this is contained in 26.048 grammes of the material used. Therefore the original percentage  $r$  of cane sugar can be calculated from the second proportion—

$$26.048 : \frac{26.048 B}{42.4 - \frac{1}{2} t} = 100 r,$$

$$\text{whence } r = \frac{100 B}{42.4 - \frac{1}{2} t}.$$

If 13.024 grammes substance were used and the solutions polarised at exactly 20° C., then the following formula is more accurate:—

$$r = \frac{100 B}{42.66 - 10} = 3.062 B.$$

III. *Liquors*.—The percentage of sugar in liquors is generally expressed as grammes per litre (say lb. per 100 gallons). Every liquor must be previously tested for invert sugar by diluting a little in a test-tube with water, adding five drops of copper sulphate solution and enough sodium hydrate to form a clear blue liquid. If no change occurs on heating, cane sugar only is present—a yellow or red precipitate indicates the presence of sugars. Liquors which do not contain invert sugar may be polarised in the 200 mm. tube, being previously decolorised with bone black if necessary. If a high percentage of sugar is present, the polarisation must be made in the 100 mm. tube. The presence of alcohol has no influence on the readings. The ethereal oils which are present, though optically active, are present in such small quantities as to be without influence. The grammes per litre R are found when A is the reading in a 200 mm. tube by:  $R = 2.6048 A$ . When invert sugar is present the alcohol must be removed, as it materially changes the rotation of this sugar.

Fifty c.c. of liquor are measured into a porcelain dish and evaporated to about one-half the volume on a water bath. If the liquid has an acid reaction, a few drops of ammonium hydrate must be added before heating it. The residue is washed into a 100 c.c. flask and made up to the mark with water. One-half is then polarised directly, the other after inversion. Both solutions may have to be decolorised with bone black.

Supposing

V = number of cubic centimetres used for analysis,

A = direct reading,

B = reading of inverted solution (both read in 200 mm. tube),

t = temperature of inverted solution when polarised;

then the grammes of cane sugar (R) in a litre may be calculated from the formula:—

$$R = \frac{26.048 (A - B)}{(142.4 - \frac{1}{2} t) V},$$

where, when the direct reading was +, the inverted reading —, the difference A - B becomes A + B.

If the temperature was exactly 20° C.,

$$R = 196.7 \frac{A + B}{V}, \text{ or more accurately } = 196.35 \frac{A + B}{V}.$$

In liquors, as under III. (b), the addition of the fruit juices may have caused inversion of a part of the cane sugar originally added. The original percentage of cane sugar is then calculated from the

inverted reading just as for candied fruits. The grammes of cane sugar ( $r$ ) per litre are found from

$$r = \frac{26.048 B}{(12.4 - \frac{1}{2} t) V}$$

or when  $t = 20^\circ$ ,

$$r = 80\frac{1}{2} \frac{B}{V}, \text{ or more accurately } = \frac{26.048 B}{32.66 V} = 797.55 \frac{B}{V}.$$

SCHEME C.—DIRECTIONS FOR DETERMINING THE BONIFICATION VALUE OF INVERT CANE SUGAR.

While the syrup is being barrelled a number of samples are taken so as to obtain a good average. This is well mixed and 250 grammes weighed out in a tared beaker. After dissolving in distilled water, the beaker is placed on the balance and enough water added to bring the weight of syrup and water up to 1,000 grammes. The syrup has then been diluted with three times its weight of water. The solution is again well mixed with a glass rod and poured into a high cylinder. A special spindle is used to determine the percentage of invert sugar. This spindle is used similarly to the Brix spindle for analysing syrups. It is graduated at  $17\frac{1}{2}^\circ \text{C.}$ , and the following table is used to correct for the temperature :—

TABLE LXXXVIII.—CORRECTION OF DENSITY OF SYRUP FOR TEMPERATURE.

Subtract from the Reading.		Add to the Reading.		Add to the Reading.	
At $10^\circ \text{C.}$	0.35° Brix.	At $18^\circ \text{C.}$	0.08° Brix.	At $25^\circ \text{C.}$	0.50° Brix.
$11^\circ \text{C.}$	0.29° Brix.	$19^\circ \text{C.}$	0.09° Brix.	$26^\circ \text{C.}$	0.57° Brix.
$12^\circ \text{C.}$	0.25° Brix.	$20^\circ \text{C.}$	0.17° Brix.	$27^\circ \text{C.}$	0.64° Brix.
$13^\circ \text{C.}$	0.22° Brix.	$21^\circ \text{C.}$	0.24° Brix.	$28^\circ \text{C.}$	0.71° Brix.
$14^\circ \text{C.}$	0.18° Brix.	$22^\circ \text{C.}$	0.31° Brix.	$29^\circ \text{C.}$	0.79° Brix.
$15^\circ \text{C.}$	0.14° Brix.	$23^\circ \text{C.}$	0.38° Brix.	$30^\circ \text{C.}$	0.87° Brix.
$16^\circ \text{C.}$	0.10° Brix.	$24^\circ \text{C.}$	0.44° Brix.		
$17^\circ \text{C.}$	0.04° Brix.				

The reading is multiplied by 4 to obtain the per cent. of invert sugar in the undiluted syrup. Hundreds are always read as an additional tenth. *Example.*—The spindle read 18.1 per cent. invert sugar at  $20^\circ$ . Hence 0.17° must be added to the reading and the sum multiplied by 4—

$$18.1 + 0.17 = 18.27 \times 4 = 73.08 \text{ or } 73.1.$$

The weight of the cane sugar used in the preparation of the syrup is then calculated by subtracting one-twentieth and multiplying the result by the weight of the invert sugar syrup. [*Conclusion of German official methods.*]

*Analysis of solid commercial glucose.*—The percentage of dextrose in crystalline glucose is determined by polarising 20.4 grammes of substance in 100 c.c. of water prepared in the same way as in the case of raw sugar and after elimination of dextrine. The degrees read on Laurent's saccharimeter give directly the percentage of glucose. With the German polarimeter 16.41 grammes of substance are dissolved in 100 c.c. of water, and the result got in a 200 mm. tube multiplied by 2, or the experiment is done in a 400 mm. tube so as to get directly the percentage of dextrose. The solution would be too concentrated with 32.8 grammes for 100 c.c., and the rotary power would no longer be constant. As with other reducing sugars, to prevent bi-rotation the solution must be boiled before being brought to the measured volume.

*Analysis of glucose syrups.*—*Composition.*—These syrups generally contain dextrose, maltose and dextrine, which are estimated by Wiley's process. 1. *Wiley's process.*—*Principles on which it is based.*—(1) The specific rotary power of these three bodies being, dextrose = + 52.74, maltose = + 138.3, and dextrine = + 194.8, a solution containing these three bodies will have a polarisation—

$$P = 52.74 D + 138.3 M + 194.8 d,$$

D, M, and d being the respective quantities of dextrose, maltose and dextrine contained in the solution. (2) If the solution be treated by mercuric cyanide in excess, the dextrose and maltose are entirely destroyed and the dextrine alone remains intact. If the solution be now polarised, the deviation observed will give the quantity of dextrine present, and we get—

$$P = 194.8 d;$$

a formula from which we can easily calculate d. Each degree of the saccharimeter being = 0.055 gramme, or = 0.089 dextrine, according as the weight taken for the polarimetric examination of cane sugar is 16.20 grammes or 26.048 grammes. (3) The difference of the two observations is—

$$P - P^1 = 52.74 D + 138.3 M.$$

The solution is then treated with Fehling's solution, which is reduced by glucose and maltose. The operation may be conducted either volumetrically or gravimetrically, and the whole is calculated as dextrose. As the reducing power of maltose is 0.65, that of dextrose being taken as unity, we get the total reduction—

$$R = D + 0.65 M.$$

By multiplying this last equation by 52.74 it becomes—

$$52.74 R = 52.74 D + 34.28 M,$$

which we subtract from 3 and we get—

$$(P - P^1) - 52.74 R = 104.02 M;$$

$$\text{whence } M = \frac{(P - P^1) - 52.74 R}{104.02}, \text{ and } D = R - 0.65 M.$$

Wiley's mercuric cyanide contains per litre 120 grammes of mercuric cyanide, 25 grammes potassic hydrate. Wiley, in testing several American glucose syrups by this method, found that the dextrose varied between 23 and 42 per cent., the dextrine between 29 and 45, and the proportion of maltose between 1 and 19. To get exact results prepare a solution of 25 grammes of syrup of glucose in distilled water. After boiling bring to 100 c.c. Polarise as above. The copper reduction is effected on a dilute solution containing 1 to 2 per cent. of the syrup to be tested.

2. *Parow's method.*—*Estimate:* (1) Dry matter in volume containing 1 gramme (if solid glucose dissolve 10 grammes in 500 c.c. of water; evaporate 50 c.c. to dryness—four hours in vacuo at 100° C.). (2) Determine ash. (3) Estimate reducing power 50 c.c., containing 1 gramme sugar, diluted to 100 c.c. and Soxhlet's method (p. 380) applied. (4) Determine reducing power after elimination of dextrine, 20 grammes of solid substance dissolved in 500 c.c., 100 c.c. drawn off (if syrup about 1 gramme is taken). Run into a 200 c.c. flask with 50 c.c. of saturated baryta water and enough 95 per cent. alcohol to bring to 200 c.c. after agitation and cooling and then filtered, 150 c.c. drawn off (3 grammes of solid glucose, 0.75 grammes of solid matter of syrup), run into 200 c.c. flask precipitated by dilute  $H_2SO_4$ , neutralised by  $NaHO$ , brought to 200 c.c., filtered and Soxhlet's method applied. (5) Determine reducing power of the inverted matter on 50 c.c. of solution diluted to 100 c.c., 10 c.c.  $\cdot 816$  (1.125) are heated on water bath for three hours, cooled, brought to 200 c.c. and Soxhlet's method applied. (6) Reducing power of inverted 100 c.c. of solution are treated with baryta water, brought to 200 c.c., filtered; 150 c.c. are treated with  $H_2SO_4$ , then 15 c.c.  $HCl$ , and heated one hour in water bath brought to 200 c.c. and filtered. This method shows that more than half the dry matter of starch syrup consists of dextrose; in solid glucose dextrine is never more than one-third of the solid matter. The percentage of sugar calculated from the reducing power is about 6 per cent. too much, because the dextrine precipitable by alcohol has a reducing power of its own. In the solid sugar there is an excess of 3 per cent. (there is less dextrine).

*Optical Determination of sugar in wine.*—The polarimetrical examination of wine is intended to discover certain sophistications, e.g., addition of cane sugar, commercial glucose, etc. The process is conducted as follows. I. *White wines.*—If the wine be a white wine, 100 c.c. of the wine are measured, and 5 c.c. of basic acetate of lead added and filtered after stirring;  $2\frac{1}{2}$  c.c. of a concentrated solution of carbonate of soda are added to  $52\frac{1}{2}$  c.c. of the filtered liquid to precipitate the excess of lead. The filtrate is examined in a 200 mm. tube, and the degrees read, multiplied by 1.1 to make allowance for the dilution of the wine tested. II. *Red wines.*—If it be a



red wine, 10 c.c. of basic acetate of lead are added to 100 c.c. of the wine and filtered, and 5 c.c. of carbonate of soda solution added to 55 c.c. of the filtrate, the mixture shaken and again filtered. The filtrate is examined in the saccharimeter, and the degrees read, multiplied by 1.2 to get the polarisation of the original wine. The wine may vary in its action on polarised light in three different ways—

1. It may be dextro-rotary.
2. It may be levo-rotary.
3. It may be optically inactive.

1. *The wine to be tested is dextro-rotary.*—It may contain saccharose or the other dextro-rotary non-fermentescible matter contained in ordinary commercial glucose. 50 c.c. are inverted with 5 c.c. hydrochloric acid in the manner already described, and a fresh polarimetric observation taken: if the inverted wine be levo-rotary, cane sugar is present; if, on the other hand, the rotation is still to the right, and exceeds that of 0.70 per cent. of glucose, it may be inferred that dextro-rotary substances from commercial glucose are present.

2. *The wine to be tested is levo-rotary.*—The left-handed rotation is due solely to inverted sugar formed by inversion of the natural sugar of the must, or by that of cane sugar added intentionally. If a previous test has been made with Fehling's solution, it is first ascertained if the polarisation be approximately equal to that which results from the presence of a corresponding quantity of invert sugar. If the left-handed rotation is appreciably inferior to the result of the Fehling's solution estimation, non-inverted cane sugar or the dextro-rotary substances of commercial glucose are present. To demonstrate the presence of the latter, the wine is fermented; if the fermented liquid be dextro-rotary, non-fermentescible dextro-rotary substances are present, originating in a fraudulent addition of commercial glucose.

*The wine to be tested is optically inactive.*—This is the case with most natural wines, and as it may sometimes be due to the simultaneous presence of dextro-rotary substances and invert sugar, so as to balance their different rotations, an inversion test is first made: if the inverted wine be levo-rotary, it may be concluded that cane sugar is present along with invert sugar. A fresh sample of the wine is then fermented, and if the fermented liquid is dextro-rotary commercial glucose is certainly present.

*The United States official method.*—This is to be determined by Soxhlet's method. The presence of unfermented cane sugar is to be shown by inversion. The wine should not contain more than 1 per cent. of sugar. *Preliminary test.*—100 c.c. of wine accurately neutralised with normal soda solution is dealcoholised by evaporation to two-thirds, and the original volume restored with water (red wines are decolorised with pure animal charcoal); 25 c.c. of copper solution and 25 c.c. of Rochelle salt solution are heated to boiling in a deep porcelain evaporating dish; wine is added from a burette till the fluid, after two minutes' boiling, no longer has a blue colour.

This gives an approximate estimate of the sugar present, and from this we can dilute the wine to very nearly 1 per cent. of sugar. *Final determination.*—25 c.c. of wine, as in preliminary test, 25 c.c. of Rochelle solution, and 23 to 24 c.c. of diluted wine (approximately 1 per cent.), are heated two minutes to boiling, the precipitated copper allowed to settle, and filtered through a double filter. If the filtrate is greenish, it contains copper; if the filtrate is yellow, it is acidulated, and, after cooling, a little acetic acid is added and two or three drops of ferrocyanide of potassium. If a reddish colour is formed, copper is still present, and a sufficient amount of sugar has not been added to reduce the 50 c.c. of Fehling's solution. The experiment is repeated, adding 1 to 2 c.c. more wine if copper was present in the filtrate; if no copper was in the filtrate, 1 c.c. less of wine. The experiment is continued till filtrates are obtained containing a difference of 0.1 c.c. of wine, one of which contains copper solution, and one not. The mean of the two contains the sugar equivalent to 50 c.c. of Fehling's solution; 50 c.c. of Fehling's solution correspond to 0.2375 gramme of grape sugar. *Polarisation.*—(1) The wine is decolorised with plumbic subacetate. (2) A slight excess of sodic carbonate is added to the filtrate from (1). 2 c.c. of a solution of plumbic subacetate are added to 40 c.c. of white wine, and 5 c.c. to 40 c.c. of red wine, the solution is filtered, and 1 c.c. of a saturated solution of sodic carbonate added to 21.0 or 22.5 c.c. of the filtrate. (3) The kind of apparatus used and the length of the tube are to be given, and the results estimated in equivalents of Wild's polaristobrometer with 200 mm. tubes. (4) All samples rotating more than 0.5 to the right (in 200 mm. tubes, after treating as above), and showing no change or but little change in their rotatory power after inversion, are to be considered as containing unfermented glucose (starch sugar) residue. (5) Rotatory power of less than 0.3 to the right shows that impure glucose has not been added. (6) Wines rotating between 0.3 and 0.5 to the right must be treated by the alcohol method. (7) Wines rotating strongly to the left must be fermented, and their optical properties then examined.

*Saccharimetric estimation of the milk sugar present in milk.*—(a) The percentage of milk sugar in milk may be determined optically. 25 c.c. of a concentrated solution of neutral acetate of lead are added to 25 c.c. of the milk to be tested, and the mixture boiled. After cooling, the bulk is made up to 100 c.c., filtered, and polarised in a 200 mm. tube. The degrees observed, multiplied by 0.4103 for French saccharimeters, or by 0.6564 for the German instrument, give the amount of sugar in 100 c.c. of the milk. The precipitate of albuminoid substances in 50 c.c. of milk occupy about 2 c.c. The bulk is made up to 102 c.c., so as to have 100 c.c. of liquid. In default of a flask graduated to 102 c.c., the result is multiplied by 0.98.

(b) *The United States official method.—The estimation of milk sugar.*

—The reagents, apparatus and manipulation necessary to give the most reliable results in milk sugar estimation are as follows: *Reagents.*

—(1) *Basic plumbic acetate*, sp. gr. 1.97. Boil a saturated solution of sugar of lead with excess of litharge, and make it of the strength indicated above. 1 c.c. of this will precipitate the albumens in 50 to 60 c.c. of milk. (2) *Acid mercuric nitrate*.—Dissolve mercury in double its weight of nitric acid, sp. gr. 1.42. Add to solution an equal volume of water. 1 c.c. of this reagent is sufficient for the quantity of milk mentioned above. Larger quantities can be used without affecting polarisation results. (3) *Mercuric iodide with acetic acid*.—KI, 33.2 grammes;  $HgCl_2$ , 13.5 grammes;  $C_2H_3O_2$ , 20 c.c.;  $H_2O$ , 64 c.c. *Apparatus*.—(1) Pipettes marked at 59.5, 60, and 60.5 c.c. (2) Sugar flasks marked at 102.4 c.c. (3) Filters, observation tubes, and polariscope. (4) Specific gravity spindle and cylinder. (5) Thermometers. *Manipulation*.—(1) Room and milk should be kept at constant temperature. It is not important that temperature should be of any given degree. Work can be done equally well at 15° C., 20° C. or 25° C. The slight variations in rotary power within the above limits will not affect result for analytical purposes. Temperature selected should be that which is most easily kept constant. (2) The specific gravity of the milk is determined by a delicate specific gravity spindle. Where greater accuracy is required, use specific gravity flasks. (3) If specific gravity be 1.026, or nearly so, measure out 60.5 c.c. into sugar flask. Add 1 c.c. of mercuric nitrate solution, or 30 c.c. of mercuric iodide solution, and fill to 102.4 c.c. mark. The precipitated albumen occupies a volume of about 2.44 c.c. Hence milk solution is really 100 c.c. If specific gravity is 1.030, use 60 c.c. of milk. If specific gravity is 1.034, use 59.5 c.c. of milk. (4) Fill up to the mark in the 102.4 c.c. flask, shake well, filter and polarise. *Notes*.—In above method of analysis the specific rotatory power of milk sugar is taken at 52.5, and the weight of it in 100 c.c. solution to read 100 degrees in the cane sugar scale at 20.51 grammes. This is for instruments requiring 16.19 grammes sucrose to produce a rotation of 100 sugar degrees. It will be easy to calculate the number for milk sugar, whatever instrument is employed. Since the quantity of milk taken is three times 20.56 grammes, the polariscopic readings divided by three give at once the percentage of milk sugar when a 200 mm. tube is used. If a 400 mm. tube is employed, divide the reading by six; if a 500 mm. tube is used, divide by 7.5. Since it requires but little more time, it is advisable to make the analysis in duplicate, and take four readings for each tube. By following this method gross errors of observation are detected and avoided. By using a flask graduated at 102.4 for 60 c.c., no correction for volume of precipitated casein need be made. In no case is it necessary to heat

the sample before polarising. *Alternate method.*—The sugar may also be determined, either gravimetrically or volumetrically, by alkaline copper solution (see Tables LXX., LXXVII., pp. 370, 385).

*Honey.*—The polarimetric examination of honey is intended to ascertain whether it be sophisticated with cane sugar or starch syrup. Biesterfeld recommends the following process: A 20 per cent. solution of honey is prepared, decolorised if need be with animal charcoal, and divided into two parts. The one is polarised as it stands, whilst the other is heated for ten minutes with a few drops of sulphuric acid, cooled, brought to the original bulk and polarised. If the honey be pure Solution I. will have a levo-rotary power of  $5^{\circ}$ , and Solution II. will also be a levo-rotary. If Solution I. deviates to the right and Solution II. to the left, then the honey is sophisticated with cane sugar; if the two solutions deviate to the right, glucose is the adulterating agent; whilst, if Solution II., although still being dextro-rotary, deviates much less than Solution I., it may be concluded that the honey is sophisticated with both cane sugar and glucose. The honey is also titrated with Fehling's solution (p. 387). It ought to contain at least 62 per cent. of reducing sugar.

*Optical determination of sugar in diabetic urine.*—The estimation of sugar in diabetic urine is easily accomplished by the optical method in the case of pale urine free from albumen. A 200 mm. tube is filled with the bright and clear urine previously filtered, if need be, and the deviation read on the polarimeter used. With the French instrument the result is multiplied by 2.049; with the German by 3.282, to get the sugar per litre of urine. If the urine in question contains but little sugar, the polarimetric observation is made in a 40 to 50 cm. tube, and the result divided by 2 and  $2\frac{1}{2}$  respectively. When the urine being tested contains albumen, which often happens, the latter must be eliminated before polarisation, the albumen being levo-rotary. To effect this, 100 c.c. of the urine are run into a porcelain basin, dilute acetic acid added, and the mixture brought to the boil. The albumen is soon deposited in large flakes, which collect on the bottom of the basin. The basin and contents are cooled, and water added to make up the original bulk, the solution filtered, and the liquid transferred to the saccharimeter. Sometimes the urine to be tested is too dark to get an accurate determination by the polariscope. 100 c.c. of the urine are then vigorously and repeatedly stirred with two or three grammes of finely pulverised animal charcoal, allowed to stand for some time, again stirred, filtered, and the decolorised liquid transferred to the polarimeter. The urine is more often decolorised by basic acetate of lead, which at the same time precipitates any small quantity of albumen present. A flask graduated in two places, namely  $100^{\circ}$  and  $110^{\circ}$  c.c., is filled with urine to the  $100$  c.c. mark, and about 5 c.c. of basic acetate of lead added and the bulk made

up to the 110° c.c. mark with water, the flask turned upside down and well shaken, the contents filtered and polarised. To make allowance for the dilution, the polarimetric reading must be increased 10 per cent. The accompanying table shows for each degree of the saccharimeter the corresponding amount of sugar in grammes per litre, the experiment being performed on a liquid clarified with 10 per cent. of basic acetate of lead in a 200 mm. tube.

TABLE LXXXIX.—TABLE FOR USE IN THE SACCHARIMETRICAL EXAMINATION OF URINE.—100 C.C. OF URINE CLARIFIED WITH 10 C.C. OF BASIC ACETATE OF LEAD.

A.	B.	C.	A.	B.	C.	A.	B.	C.
1	2.25	3.61	6	13.50	21.66	11	24.75	39.71
2	4.50	7.23	7	15.75	25.27	12	27.00	43.32
3	6.75	10.83	8	18.00	28.88	13	29.25	46.93
4	9.00	14.48	9	20.25	32.49	14	31.50	50.54
5	11.25	18.05	10	22.50	36.10	15	33.75	54.15

A = Degrees observed in 200 mm. tube. B, Grammes of sugar per litre of urine, French saccharimeter. C, Grammes of sugar per litre of urine, German saccharimeter.

*Analysis of jam—detection of glucose.*—1. Twenty grammes are dissolved in 200 c.c. of water and the solution halved. One half is inverted by a few drops of sulphuric acid, and the cane sugar and invert sugar estimated by Fehling's solution. 2. Five c.c. of basic acetate of lead are added to 50 c.c. of Solution I., filtered and polarised. After inversion the deviation should be to the left. From this deviation the amount of invert sugar is calculated. It should fairly correspond with that found with Fehling's solution. 3. If after inversion the liquid deviates to the right, the jam contains glucose, the presence of which is confirmed as follows: 200 grammes of the jam are dissolved in a litre of water and fermented as long as CO<sub>2</sub> is evolved, the liquid is then dialysed, renewing the dialysed liquid daily until it shows no further deviation, i.e., until it contains no more sugar. The liquid remaining is decolorised by bone black and polarised. If a deviation to the right be observed, and the liquid when tested by Fehling's solution shows no glucose, and the dextro-rotary substances are insoluble in absolute alcohol, the original jam contains glucose, as this reaction is characteristic of the accessory amylaceous products present in commercial glucose.

The Syndical Laboratory of Berlin, Z.V.D., 1903 analysed jams and marmalades of British exportation, or purchased in Britain itself, to determine *inter alia* whether British makers use a certain proportion of starch sugar to prevent crystallisation. Dejeners says they add 10 per cent. The results, except in small amount in

isolated cases, were negative as well as those to detect preservatives, thickenings and colourings; but these samples can hardly have been very representative as they seem to be unaware that British jam manufacturers also make jellies—the only German form. The use of glucose in Germany is universal and often carried to excess.

*Estimation of sucrose in preserved bananas.*—Munson and Tolman analysed (a) 38 samples of fresh bananas, 21 from Florida, 10 from Cuba, 4 from Porto Rico, 2 from the Bahamas and 1 from Jamaica; (b) 16 samples of preserved bananas bought by U.S.A. Consul-General at Singapore (10 in the juice pressed from the bananas, 6 in the pressed juice with sugar added), 2 samples from Nassau; (c) 42 samples of commercial preserved bananas from Singapore, Straits Settlements and Bahamas.

*Analysis.*—Dry matter is determined by drying in an asbestos stove for thirty hours. The dry matter of the syrup is deduced from its density by Ellion's table. Reducing sugar is estimated by Meissl's method and sucrose by the increase of the reduction after inversion with hydrochloric acid and double polarisation. In the latter case Herzfeld's formula is used—

$$S = \frac{100 A - B}{141.89 + 0.05 B} - \frac{x}{2}.$$

The two methods gave concordant results if there was little sucrose. If much, it affected the reduction. The acidity of the bananas was due to citric acid but is expressed as  $H_2SO_4$ .

The following results were obtained :—

TABLE XC.—ANALYSES OF BANANAS, FRESH AND PRESERVED.

	Fresh Bananas.			Preserved Bananas.					
				Without Addition of Sugar.			With Addition of Sugar.		
	Min.	Max.	Aver.	Min.	Max.	Aver.	Min.	Max.	Aver.
Total dry matter .	10.78	18.86	14.17	10.00	17.44	13.27	16.61	19.11	18.17
"    "    " insol.	1.02	1.83	1.52	0.87	2.18	1.37	1.02	2.06	1.44
Total ash .	0.27	0.55	0.40	0.24	0.56	0.39	0.27	0.51	0.40
Alkalies as $K_2CO_3$ .	0.22	0.60	0.37	0.21	0.66	0.33	0.16	0.34	0.23
Acids as $H_2SO_4$ .	0.30	0.85	0.60	0.25	0.71	0.42	0.20	0.42	0.32
Protein ( $N \times 6.25$ ) .	0.21	0.57	0.42	0.25	0.57	0.46	0.35	0.46	0.41
Reducing sugar .	1.76	9.75	3.91	5.44	10.96	8.00	7.51	15.39	11.63
Sucrose .	2.98	10.48	7.59	1.85	6.61	3.40	2.28	8.82	5.14
Total sugar as invert sugar .	8.20	15.28	11.90	8.55	14.89	11.59	16.70	17.93	17.41

TABLE XCI.—ANALYSIS OF COMMERCIAL PRESERVED BANANAS.

	Singapore and Straits Settlements.			Bahama Isles.		
	Min.	Max.	Aver.	Min.	Max.	Aver.
Total dry matter . . . . .	17.32	26.84	21.03	8.54	26.78	14.13
Total dry matter insoluble . . . . .	0.91	1.67	1.14	0.68	2.51	1.39
Total ash . . . . .	0.21	0.96	0.27	0.22	0.50	0.33
Alkalies as $K_2CO_3$ . . . . .	0.14	0.32	0.22	0.20	0.39	0.30
Acids as $H_2SO_4$ . . . . .	0.16	0.43	0.26	0.22	1.18	0.56
Protein ( $N \times 6.25$ ) . . . . .	0.39	0.57	0.46	0.20	0.46	0.33
Reducing sugar . . . . .	7.05	15.18	9.91	5.55	12.84	7.96
Sucrose . . . . .	4.34	16.48	7.77	0.53	9.05	2.78
Total sugar as invert sugar . . . . .	14.55	25.10	18.04	6.33	22.37	9.98

The fresh bananas show great differences in sugar-content. Generally there is twice as much sucrose as reducing sugar. There is little variation in dry matter, ash, acids and protein. Bananas preserved without sugar are generally preserved in 30 per cent. of their juice. Their composition should be much the same as fresh bananas, only there is relatively less reducing sugars and much less cane sugar. The addition of juice, moreover, reduces the insoluble. The same occurs with bananas preserved with sugar; the samples of the latter from the Straits Settlements must have had sugar added, the Bahamas samples partially. Wiley concludes that for customs purposes all samples containing less than 14 per cent. of total sugar in each can and a maximum of 13 per cent. over a whole cargo, may be regarded as bananas preserved in their juice, and that the bananas have been preserved with sugar when they contain more than 14 per cent. in a tin, or an average of 13 per cent. on a whole cargo.

## CITY GUILDS EXAMINATION IN SUGAR MANUFACTURE.

## PROGRAMME.

1. *Syllabus*.—The Examination will include questions founded on such subjects as the following:—

*Ordinary Grade*.—1. Cane sugar: its occurrence in nature. 2. Methods of extracting the juices of sugar-yielding plants; composition of the juice so extracted. 3. The principal methods of making raw sugar from these juices and the apparatus employed, including the "vacuum pan," "multiple-effect pan" and "concretor". 4. Refining of sugar. Practical details of melting and bag filtering and boiling in vacuo. 5. Treatment of the boiled mass for the preparation of "loaf sugar," "crystals" and "pieces" (crushed) respectively.

6. Treatment of the syrup to secure the extraction of the last available portions of sugar. 7. Molasses or treacle, composition and nature of. 8. Animal charcoal; its preparation, composition, revivification and action on cane and beet juice and solutions of raw sugar. 9. The centrifugal machine, general construction of some of the principal forms of—method of using, explanation of its action. 10. Apparatus and machinery in general use in sugar factories and refineries.

*Honours Grade.*—In the Honours Examination more difficult questions will be set in the above subjects, and in addition a knowledge will be required of—1. Analysis and valuation of raw sugar and syrups, construction and principles of the instruments and the preparation and standardising of the re-agents employed. 2. The chemistry of cane sugar: its composition, properties and its changes under the action of heat, acids and "ferments". 3. "Glucose"—its properties and its relation to cane sugar, distinguishing between dextrose and levulose. 4. The chemistry of animal charcoal. 5. The physics of the vacuum pan (involving properties of steam).

II. *Full Technological Certificate.*—A Provisional Certificate will be granted on the results of the above Examination. For the Full Technological Certificate in the Ordinary Grade, the candidate who is not otherwise qualified (see Rules 40-41) will also be required to have passed the Examination of the Board of Education in the Elementary Stage at least; and for the Full Certificate in the Honours Grade, in the Advanced Stage at least in two of the following Science subjects:—

- II. Machine Construction and Drawing.
- VI. Theoretical Mechanics.
- VII. Applied Mechanics.
- X. Inorganic Chemistry.
- XI. Organic Chemistry.

## SPECIMEN OF EXAMINATION PAPER.

### ORDINARY GRADE.

*Examination Questions.*—1. Give the formulæ and molecular weights of cane sugar, dextrose and levulose. 2. Describe the nature and use of the apparatus known as a montejus. 3. Name the constituents of new char which you consider important in determining its value. 4. Describe an ordinary centrifugal machine. 5. Which is more suitable for refining sugar, soft or hard water? Give reasons for your answer. 6. Name, in the order of their relative importance, four of the chief sources of sugar. 7. Give a short account of the manufacture of sugar from the sugar cane, including the use of vacuum pans and centrifugals. 8. What are the chief impurities in raw beet and cane sugar respectively.



## HONOURS GRADE.

1. State the approximate temperature at which char should be reburned, and how you would ascertain if the char had been over or underburned. 2. Explain the use of blood, albumen and phosphoric acid as defecating agents in the blow-up. 3. If it were considered desirable to raise the quality of a grainy sugar before melting for refining purposes, in what manner would you accomplish this? 4. Give the mode of preparing Fehling's copper test, and of employing the same. 5. What are the differences between the hydro-sucro-carbonate processes of Boivin and Loiseau, or Tooth's, and the carbonatation process as carried out in a beet-sugar factory? 6. If the products of a refinery were contaminated with iron, in what way would you mitigate this evil? 7. Give a short account of one of the methods for producing cube sugar from centrifugal sugar. 8. How would you arrest, without the use of chemicals, fermentation in a solution of cane sugar? 9. The specific gravity of a 50 per cent. solution of cane sugar is 1.2327; how much sugar in the form of such a solution could be contained in a tank of the following dimensions: 12 ft.  $\times$  8 ft.  $\times$  4 ft.?

On the results of above examination held annually in April are awarded the—

SALTERS' COMPANY'S PRIZES IN SUGAR  
MANUFACTURE.

## HONOURS GRADE.

A Prize of £2 and Institute's Silver Medal.

## ORDINARY GRADE.

1st Prize . . .	£1 and Institute's Silver Medal.
2nd „ . . .	£1 „ „ „ Bronze Medal.
3rd „ . . .	Institute's Bronze Medal.

N.B.—Candidates resident in India or Colonies desiring to be examined should apply to their Director of Public Instruction. Home candidates to the nearest Technical Institute, or to the Superintendent, Department of Technology, City and Guilds of London Institute, Exhibition Road, London, S.W.

## INDEX

ACCELERATION of centrifugals, 203.

Acetone, 345.

Acetous fermentation of cane juice, 275.

Achard (defecation), 70.

Acidity of cane juice, 276; — cane sugar, 235, 286.

Acids: acetic, 276, 343-45; aconitic, 237; benzoic, 339; butyric, 73, 344; carbolic, as froth preventer, 193, 194; citric, 74, 237, 425; gluconic, 238; glycollic, 237; hydrochloric, in bone black purification, 114, 307, 308; lactic, 73; malic, 74, 343; mucic, 352; nitric, 74, 344, 347; oxalic, 74, 347, 352; pectic, etc., 72; phosphoric, 71; saccharic, 238, 352; saccharinic, 238; silicic, 74; stearic, 344; sulphosaccharic, 347; sulphuric, 70, 310; sulphurous, 230-32, 277-80, 297; tannic, 237, 345, 352, 412; tartaric, 343.

Air-pumps, 161-64, 308-11.

Albumen in beet juice, 72; — in cane juice, 238, 275, 276.

Alcohol, 348; — industrial, from cane molasses, 231; — solubility of glucose in, 347; — of sucrose, 343.

Alcoholic extraction (analytical) of sugar from beets, 20; — of sugar from molasses, 225-28; — fermentation of cane juice, 275.

Aldehyde, 345.

Alkalinity of carbonated juice, titrating, 79, 97; — limed juice, titrating, 97; — of raw beet sugar, 285-6.

Alum and aluminium hydrate in sugar estimation, 412-15.

Alumina and lime, superphosphates of, in refining, 290, 291.

Ammoniacal gases of juice, pumps, taps and receivers for elimination of, from carbonatation triple-effect, etc., 93, 153, 183.

Amylaceous matter (maltose), 351.

Analytical results of analysis of: bananas, fresh, 425, preserved, 426; beets, 71; beet juice, 71; beet pulp (spent slices), 60; bone char, new, 295; old, 306; *masse cuites* (sugar-cane diffusion), 231; molasses, 251, 281, 396, 403, 404; raw sugars, 284.

Analytical schemes for valuation of sugar content of: bananas, 425, 426; beets, 9-24; beet juice, 12, 72; beet juice carbonatation sludge, 106; beet pulp, 14-19; beet pulp (spent slices), 63-65; beet syrup (triple-effect) and *masse cuites* vacuum pan, 194, 195; beet juice, weak liquors, 61-65; cakes (pastry), 414, 415; candies, 414, 415; chocolate, 414; compots, 415; confectionery, 414; dessert bonbons, 414; dragees, 414; fondants, 414; glucose, 418, 419; honey, 423; jams, 424; jellies, 415; liquors, 416; marzipan, 414; milk, 421; pastry, sweetened, 414; pralinees, 414; preserved fruit, 415; raffinade wafers, 414; santonin worm wafers, 414; sugar cane, 238-74.

Animal Charcoal. See Bone Char.

Antimonic chloride (on sucrose), 344.

Ash in *masse cuites*, 194; in sugar, 388-91; in syrup, 194.

Automatic bagging up of sugar, 220-23; — burette, 78, 99; — discharging diffusion battery, 65, 66; — juice registers, 82-83; — samplers, 158, 176; — sugar mixer, 221; — weigher, 221.

BAGASSE, 249-55; — and molasses (mixed) as cattle food (molasscuit, etc.), 252-55; — furnace, 252-3 (see cane-diffusion chips, spent); — levigation, systematic, of, 249-52.



- tanks, 80; — gas scrubbers, coolers and pump, 95, 96; lime gauging tanks, 97; liming tanks, 78; *diffusion plant*: batteries, automatic discharge, circular and line, 51, 53, 57; calorimeters, 39; cells, 51; gas eliminator, 56; juice measuring vessels, 88; — registers, 88-89; — stray pulp eliminators, 68; *filtration plant*: filter presses, 102-9; filters, bag, 119; — bone black, 102-18; — mechanical, 119-23; — sand, 119; montajus and filter press pumps, 104; triple-effect evaporation to syrup plant, 124-76; vacuum pan evaporation of syrup to strike point, 177-201.
- Beet pulp (for beet analysis), 15.
- Beet pulp, spent (diffusion slices), 57-67.
- Beet seed, cost of, 2-7.
- Beet seed producing plants, testing, 18-20; — — cost, 20.
- Beet-sugar factory, cost of installing, equipping and working, 6, 7.
- Beets, analysis, direct, 14; — indirect, 12-14; — cable conveyance of, by, 25; — cartage of, farm to factory, 1-7; — heading and tailing, 8; — purchasing, 7; — sampling, 8; — preparing for analysis, 10; — steam and coal consumed in treating ton of, 125-28, 130, 199; — storage of, 8; — tare, 8; — water mill lade, transport of, by, 24, 25.
- Benzoic acid (sugar ash process), 389, 390.
- Bessemer cane-juice expresser, 248-9.
- Biosas, 337, 339.
- Blending sugars, 220-23.
- Boiling beet juice to syrup, 124-76; — beet syrup to strike-point, 177-201; — cane syrup to strike-point, 272-81; — sugar (raw) liquors defecated to strike-point in refining, 308-35.
- Bonbons, 414.
- Bone char, manufacture, 111, 112; — — use in beet-sugar works, 109-19; — — — revivifying, 114-19, 302-8; in treating cane diffusion juices, 267; in sugar refining, char per ton of sugar, 305; — — new and old or used composition, 295-306; nitrogen in, 295; size or grist, 299, 300.
- Bonification value of invert sugar, 417.
- Brown sugar, 216-19.
- CALANDRIA of vacuum pan, 182.
- Calorie, 127.
- Calorimeters, beet diffusion, 39, 53; — cane diffusion, 260.
- Candies, analysis of, 414.
- Candy, sugar-crystals, 342.
- Cane Sugar. See Sucrose.
- Caramel, 335, 336, 344, 347.
- Carbonatation, 69-109. See Beet Juice.
- Centrifugals. See Turbines.
- Cerosin, 237, 238.
- Chocolate, determination of sucrose in, 413.
- Circular diffusion batteries, 51, 53, 57, 260.
- Clarification. See Defecation and Lead Acetate (basic), etc.; — during diffusion, by lime, 265.
- Clerget's formulae, 399; table, 400, 401.
- Coal consumption in beet-sugar works, 125-28, 130, 131, 170, 270, 271; in cane-sugar diffusion versus milling, 270, 271; in triple, etc., effect evaporation versus single-effect, 131, 148, 170; in vacuum strike pan evaporation, 199, 201.
- Colloids of beet juice, 85, 96.
- Compots, determination of sucrose in, 414.
- Condensation vacuum, 308.
- Condense box (vacuum pan), 317.
- Condensed water, 158.
- Condensers, 161-65, 313, 319; — air-pumps for, 163, 311; — barometrical, 163, 164; — capacity, 165; pressure, 165.

- Confectionery, determination of sucrose in, 414.  
 Continuous carbonatation of beet juice, 89-92; — turbinating of sugar, 206.  
 Coolers, crystallisation, in motion, 186, 190, 210.  
 Coolers, reheaters in sugar refining, 185, 190.  
 Corrosion in boilers from dissolved sugar, 161.  
 Creydt's formulae, 399.  
 Crystallisation in motion, 186, 190, 232.  
 Crystalloids, 35, 86.  
 Cube sugar, 333-35.
- DAHLIA, inulin in, 349.  
 Decomposition products of sugar (dry distillation), 344, 345; — — fermentation, 337, 340, 343.  
 Decrepitation in vacuum pan (crystals, formation of), 320.  
 Defecation. See Carbonatation (beet juice), Lead Acetate, etc.  
 Defecation, cane juice, 275-82.  
 Density of beet juice, 11; cane juice, 274; sucrose, 12, 178, 180, 339, 341.  
 Dextrine, 350, 351.  
 Dextrose. See Glucose.  
 Diabetes, glucose in, 423.  
 Dialysis, 34-6, 225-7.  
 Diffusion, of beet juice, 38-68. See Beet Juice, Beet Machinery and Sugar-cane Juice; — temperature in beet and sugar-cane cells of diffusion battery compared, 267.  
 Dilution of diffusion juice (beet), 131; — — (cane), actual, 268, 269; — — apparent, 268, 269.  
 Dissolved matter of juice (beet), 14; — matter of carbonated juice, 101; — — *masse cuite*, 193; — — of raw syrup, 193.  
 Distillation, dry, of sugar, 344, 345.  
 Dragees (sucrose in), 414.  
 Dry distillation sugar, 344, 345.  
 Drying sugar, 220.  
 Drying washed beets, 28, 29.
- ELEVATORS, beet, 29-31; sugar, 221.  
 Eliminators: pulp from juice, 67, 68; stones from limed juice, 101, 102; stones from beets, 25-28.  
 Encrustation, effect on heat conduction of triple-effect, 152; on Yaryan, 175; remedy, 175.  
 Evaporation defined, 124; — horizontal systems, 137-40, 179; Lillie's system, 175; multiple-effect, 124-76; quadruple, 150-66; quintuple, 150-67; nonuple, 166; single effect streaming system, 175; vertical systems, 138-40, 181; Yaryan, 175; Wetzells and Chenaller's system, 280.  
 Evaporation tables, juice to syrup, 125, 128, 130, 131, 186, 141-48, 158, 163, 170, 171; syrup to *masse cuite*, 199-201.
- FALL of heat in consecutive vessels of multiple-effect, 140, 150-52.  
 Fall pipe (condenser), 164.  
 Farm labour, cost of, in raising beets, 2-7.  
 Farmer as shareholder in beet factories, 1, 6.  
 Fermentation of beet juice, butyric, 73; lactic, 73; pectic, 72; secondary, 73.  
 Fermentation of bioses sucrose, 337-39, 344; monoses (glucose), 337-39; maltose, 351.  
 Fermentation of cane juice, acetous, 275, 276; — — alcoholic, 275, 276; — — syrup in storage tanks, 272.  
 Fill-houses, 216.  
 Filter presses, 102-9.  
 Filters, bone char, 102-13; — cloth bag, 119; — mechanical, 119-23; — sand, 119.

- Filtration of beet juice, 97-123. See Beet Machinery; — cane juice, 273-77;  
— raw sugar liquors in refining, 291-303.  
Froth breaking in carbonatation, 159; — — triple-effect evaporation, 159.  
Fruit sugar, 337, 343, 345, 349 (*syn.* glucose (dextrose), and levulose in molecular proportions, *which see*).  
Fruits, preserved (sucrose in), 415.
- GALACTOSE, 352, 399.  
Gas (CO<sub>2</sub>). See Kiln, Lime, and Beet Juice Carbonatation; — engine, — steam, consumption of, 200; — juice elimination from triple-effect, 172.  
Germany, beet culture in, 3; cost of beet-sugar factories in, 6, 7.  
Glucosane, 347.  
Glucosates of baryta, 348; — lead, 348; — lime, 348.  
Glucose, dextrose, 345-49, 351, 352, 364-68, 369, 370, 378, 380, 381, 386, 387, 403, 405, 406; — — analysis (scheme), 418, 419.  
Glucosides, 348.  
Grape sugar. See Glucose, Fruit Sugar.  
Gravimetric analysis of raw sugar, 374-77, 380-86.
- HANRIOT's beet pulper (analysis), 16.  
Heat radiators, 92, 93, 161, 183, 184.  
Heating by direct fire, 125, 126.  
Heating by steam, 126 *et seq.*; — — open steam injurious, 288.  
Heating surface, 130, 142.  
Hodek (froth and vesicular sugar curb), 159.  
Honey, 337, 345, 346, 349, 388, 423.  
Howard's vacuum pan, 124-27, 131-33, 135-37, 306-29.  
Hughes' cane cutter, 257-58.  
Hydration products (starch), 351.
- INJECTION condenser, 318.  
Injection of CO<sub>2</sub> into limed beet juice, 78.  
Injection of steam and air into turbine for cleansing raw sugar, 212.  
Invert sugar and inversion, analytical schemes and processes for, 374-79, 382, 383, 386, 392, 393, 395, 398, 403-9, 415-20, 424-26.  
Invert sugar, syrup correction for temperature, 417.  
Iron in raw sugar syrups, 306, 307.
- JAMS, 415, 424.  
Jelinek's evaporation data, 142-47.  
Juice. See Beet Juice and Sugar Cane Juice.
- KILNS, lime, (beet-sugar works), 96, 97.  
Knives of beet slicers, 41-49; — cane cutters, 257.  
Konig's (vesicular) sugar recoverer, 160, 161.
- LABORATORY (beet-seed producer, testing), 17-20; — (polariscope work), 355.  
Lactic fermentation, 353; — beet juice, 73.  
Lactocaramel, 352.  
Lactose. See Milk Sugar.  
Lead acetate, basic (as clarifier in sugar analysis), 12, 13, 17, 18, 20, 21, 23, 61, 64, 65, 72, 85, 87, 103, 194, 239, 297, 412, 413, 414, 421, 423; — as defecating agent in sugar refining, 297; — debarred by toxic nature, 297.  
Levulosane, 344.  
Levulosate of lime, 349.  
Levulose, 349, 351, 370, 387. See also Invert Sugar.  
Lime, milk of, 97.  
Limestone, analysis scheme, selection for burning in beet-sugar works kilns for liming beet juice, 94, 95.

- Line diffusion batteries, 51, 53, 57.  
 Lignoring, claying sugar loaves, 329-33.  
 Liquors, determination of sugar in, 416.
- MACERATION**, beet-juice extraction, 33.  
 Maltose, 350, 351.  
 Mannite, 353.  
 Manoury sugar recovery (molasses), alcoholic, 225-28.  
 Marmalade, sucrose in, 413.  
*Masse cuite* (beet) alkalinity, 195; analysis (scheme), 194; ash, 194; dissolved matter, 195; lime (total), 195; non-saccharine, 195; purity, 195; saline coefficient, 195, 196; sucrose estimation, 194; — composition of different densities, 197; — total crystals in, 195; — engine, steam, consumption, 201; hectolitre of syrup required for, etc., 201.  
 Mechanical filtration, 101-9. See Filters.  
 Melada, 388.  
 Melezitose, etc., 387, 388.  
 Milk sugar, 337, 338, 351, 354, 363, 370, 385-87, 421-23.  
 Mills and milling, sugar cane, 242-53.  
 Molasses, analysis, 395-404; beet, 224; — recovering sugar from, 224-30; — by alcohol, 225-28; — by lime separation, 226; — by osmogène, 225-27; — — sucrate separation process, 227, 228; — strontia separation, 230; — sugar content of, 224; — lost in, 224; — cane sugar and molascuit, 281.  
 Monoses, 337-39.  
 Montejus, 104, 289, 328.  
 Moulds (loaf), 329-33; cube sugar, 382-35.  
 Multiple-effect. See Evaporation.
- NICOL prism analyser, 355-60.  
 Nitric acid, action on sucrose, 314; — — — dextrose, 348;
- OPEN pan evaporation, 125-28; — steam, over-heating by, 288-9.  
 Optical rotation of sugars, 354, 362, 363.  
 Osazones, 349.  
 Osmogènes, 225-27.  
 Osmosis, 35, 36, 225-27.  
 Ozone. See Beanes.
- PECTIC** fermentation (beet), 72, 73.  
 Pectic and pectose bodies (beet), 71-73.  
 Pellet's digestion processes in beet analysis, 14-19; — pulping machine (beet analysis), 14-17.  
 Perichon's bagasse levigation process, 249-52.  
 Polariscopes. See Saccharimeter.  
 Pressure, beet-juice extraction by, for analysis, 18-12; — — for manufacture (defunct), 32.  
 Pulp, beet (spent slices), 57-65; — — — pressing, 57-60; — — — testing, 59, 60; — — — value, 60.  
 Purity of carbonated juice, 101; of syrup and *masse cuite*, 194, 217-19.
- QUADRUPLE-EFFECT**, 161.\*  
 Quarex beet-juice sulphitation, 330-32.  
 Quintuple-effect, 166, 167.
- RADIATING** heater, 92.  
 Refined wafers, sucrose in, 414.  
 Raffinose, 387, 388; determination of, in sugars, syrups and molasses, 399, 401, 405, 409, 410.  
 Raw sugar, activity of cane, 285, 286; — alkalinity of beet, 285; — analysis scheme, 337-95; — — results of, of different brands, 284; — liquors in refining, defecation of, 287-91; — — density of, for refining, 289; —

- defecated filtration, 291-308; — — — evaporation of, to strike-point, 308-29; — — — turbinizing of, as substitute for refining, 286.
- Reagents and heat, action of, on glucose, 249-49; lactose, 351, 352; levulose, 349, 350; maltose, 350, 351; sucrose, 342-45.
- Registers, juice (beet) diffusion, 83, 88; — — (cane), diffusion, 261-63.
- Reheating juice in carbonatation tanks, 78, 92, 93; — — — diffusion calorimeters, 99; — — — maceration vats, 33.
- Reheating *masse cuite*, 190.
- Revivifying bone char in beet-sugar works, 114-19; in sugar refining, 302-8.
- Rillieux system of evaporation, 127-75.
- Rotation, optical, of sugars, 354, 362, 363.
- SACCHARIDE, 344.
- Saccharimeters, 353-64; — French, 354; — German, 354.
- Saccharimetrical constants of sugars, 354, 362, 363.
- Saccharimetrical flasks, graduation of, 361.
- Saccharimetrical valuation of sugars and saccharine juices. See Analytical Schemes, etc.
- Safety vessel, 158-61. See Hodek, König.
- Salicin and saligenin, 348.
- Sampling beets, 8-10; — beet slices, 50; — beet juice from diffusers, 85-87; — sugar canes, 238; — syrup, 158, 176.
- Santonin wafers, sucrose in, 414.
- Scoffern's lead acetate defecation process, 297.
- "Seconds" sugars, 215, 216, 230, 233, 235.
- Seed, beet, cost of, 3-6; — — — producers testing, 18-20.
- Seed, sugar-cane, propagation of, from, 236; separation methods of recovering sugar from molasses, 227-34.
- Siloing pressed spent pulp, 60.
- Silos, beet, 23, 24.
- Slicing machinery. See Knives, Beet Machinery and Cane Cutter.
- Sludge from carbonatation of beet juice analysis, 107; filter pressing and washing, 101-8; sugar lost in, 106, 109; manurial value, 107.
- Sludge pump engine, steam consumption, 200.
- Soda crystals and baryta carbonatation with, 84.
- Soldaini's reagent, 371-76.
- Solubility in water of glucose, 347; levulose, 350; sucrose, 178, 180, 342; — in alcohol of glucose, 347; sucrose, 342.
- Sorhlet's gravimetric method, 380.
- Starch, 345-6; — hydration, products of, 350-52.
- Steam in beet sugar factories, comprehensive tabular data, 199-201. See also Evaporation, etc.
- Steam, latent, heat of, 132.
- Steffen's separation methods, 227, 228, 280.
- Stocktaking, 195.
- Stone eliminators from beets, 25-28; from limed juice, 101, 102.
- Strontia as a defecating agent, 82; — — — a separating agent in sugar recovery from molasses, 230, 335.
- Sucrate of lime, molasses separation process, 227, 228.
- Sucrate of strontia, 230.
- Sucrates of lime, 73-75, 227, 228.
- Sucrocarbonates of lime, 78-75.
- Sucrose, 337-45; — density of solutions of, 12, 178, 180, 339, 341; optical rotation of, 354, 362; solubility of, 178, 180.
- Sugar, action of heat and reagents on, 343; — analysis of raw, scheme, 387-95; bagging up, 220-23; blending, 220, 285; — blow up pan for dissolving raw, for refining, 287-91; — boiling, 309-35; — — in grain, — — — point of solutions of, 180; — — — to string proof, 272; — brown, 216-19; — crystallisation of, 342; — — in motion, 186, 190, 232; — cube, 333; — distillation, dry, of, 344, 345; — drying, 220; — fermentation of, 337-39; — gravimetric analysis of, 393; — hard water in refining, 304; — invert, 374;



- liquors, defecation and decolorisation by: basic acetate of lead, 287; by ozone, 296; by sulphurous acid, 297; non-applicability of double carbonatation to, 298; — density for refining, 281; — filtration through bag (Taylor's) filters, 291-94; — — — bone black, 294-99.
- Sugar cane, bagasse, extraction of last trace of sugar by levigation, 249-52; chips, 257; — — exhausted, 254-56, 260; — cultivation, 239-42; — juice, acetous fermentation of, 276; — — alcoholic fermentation of, 276; — boiling to strike-point, 281; — — coal consumed in boiling to strike-point versus beet juice, 269-71; — — composition of, 237, 238; — — defecation of, 265, 266, 275-80; — — dilution due to diffusion, 268-70; — — evaporation of, 281; — — extraction by diffusion, 254-72; — — — milling, 242-48; — — — percussion pressure, 248, 249; — — liming, 276; — — liming during diffusion, 265, 266; — — liming plus sulphitation, 277; — — milling, 242-53; — sulphitation, 277.
- Sugar-cane machinery, bagasse levigation plant, 249-52; Bessemer's juice expresser, 248, 249; chip chute, 255; cooler (*masse cuite*), 282; cutters, shredders, etc., 244, 245, 257-59; defecation pan, 282; diffusion batteries, 254-73; — — calorimeters of, 260; — cell mills and milling, 242-53.
- TABLET, sugar (cube), 303-26.
- Tanks, bone black, 294, 299, 301.
- Tanks, carbonatation, 80, 81, 88-92; — liming, 78; — measuring (juice), 83, 84; — reheating.
- Taylor's bag filters, 119, 291-94, 301.
- Temperature, fall of, in consecutive triple-effect vessels, 141, 151, 152.
- Temperature in beet versus cane diffusion, 267.
- Triple-effect apparatus. See Evaporation.
- Triple-effect engine, steam, consumption of, 200.
- Triple-effect, horizontal, 137-40; vertical, 138-40.
- Tube fixing in Austrian multiple-effect plant, 175.
- Turbines, centrifugals, 202-23; American, Austrian, 204; French, 206; — suspended, 205; — Weston's, 205; — continuous, 206; — acceleration of, 203; — centrifugal force of, 202; — cleansing of sugar in, by steam injector, 212; — feeding of, 209-10; — feeding by overhead trucks, 209, 210; — history of, 203; — types of, 204; — speed of, 203; working of, 203.
- Turbine engine, steam consumed by, 200.
- UNCRYSTALLISABLE sugar. See Fruit Sugar.
- Urine (glucose estimation), 423.
- VACUUM pan, calandria of, 182; — coils of, 183; — lyres of, 182; — radiating heaters of, 183.
- Vacuum pan, Howard's, 309; — testing, 315; — working, 309-29.
- Vacuum pan, Lexas', 179; — types, vertical, 181; horizontal, 309.
- Violette (volumetric analysis), 364-68.
- Volumetric analysis of sugars, 364-74.
- WASHING machines, beets, 25-28; — bone black, 114-17; — cane bagasse, 249-52; — sludge, beet, 105, 109; — sugar leaves, liquoring, 323-33.
- Weighing machines, beets, 30-32; — sugar, 221.
- Wine determination of sucrose in, 419.
- Witkowitz's radiating heater, 161.
- YABYAN multiple-effect evaporator, 175, 263.
- Zinc oxide (sugar ash determination by), 389.

THE END

*Advertisement Sheet.]*

The following pages show some of the many designs  
of Centrifugal Machines which we have supplied.

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# CENTRIFUGAL MACHINES.

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PATENTEES AND SOLE MANUFACTURERS:

**WATSON, LAIDLAW & CO.,**

98 DUNDAS STREET, GLASGOW, SCOTLAND.

(1)



# WATSON, LAIDLAW & CO.,

98 DUNDAS STREET (SOUTH), GLASGOW, SCOTLAND.

MAKE  
**CENTRIFUGAL MACHINES,**  
DRIVEN BY ELECTRICITY, WATER OR BELTING,  
**ROTARY PUMPS,**  
**CONVEYORS,**  
**ELEVATORS,**  
AND ALL REQUISITES FOR  
**CURING SUGAR,**  
INCLUDING  
**CRYSTALLISATION IN MOTION PLANT.**

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SPINDLES WITH PATENT BALL BEARINGS.

SPINDLES WITH PATENT RUBBER BUFFER.

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PATENT SPINDLES	...	...	...	...	...	...	pages 4 and 5
CENTRIFUGAL MACHINES, driven by Electricity	...	...	...	...	...	...	6-8
"	"	"	Water	...	...	...	10-12
"	"	"	Belting	...	...	...	16-18
CRYSTALLISATION PLANT	...	...	...	...	...	...	page 20

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**PATENTEES AND SOLE MANUFACTURERS—**  
**WATSON, LAIDLAW & CO., Engineers, Glasgow.**  
 (3)

## IMPROVED BALL-BEARING SPINDLE

WITH

### PATENT CONOIDAL BUFFER.

---

THE advance which has been made in recent years in the manufacture of ball bearings, has enabled us to design a combination of ball bearings which not only acts as a thrust bearing to carry the load, but also to do without brass bushes to take the side pressure. This being so, the spindle may be made solid and in one piece—all bushes and other parts common to the "Weston" spindle, or any modifications thereof, being dispensed with.

A feature in the "Weston" spindle was the provision for lubricating; this is not necessary with our improved solid spindle, for there are no bushes, and the ball bearing only requires a little motor grease once or twice a year.

The particular form of our **Improved Patent Buffer** is a most important departure in the suspension of the "Weston" machine. It retains all that has hitherto been a feature in any form of buffer, but it does much more; for, by its use, the basket will run with a much greater unbalanced load than has hitherto been possible.

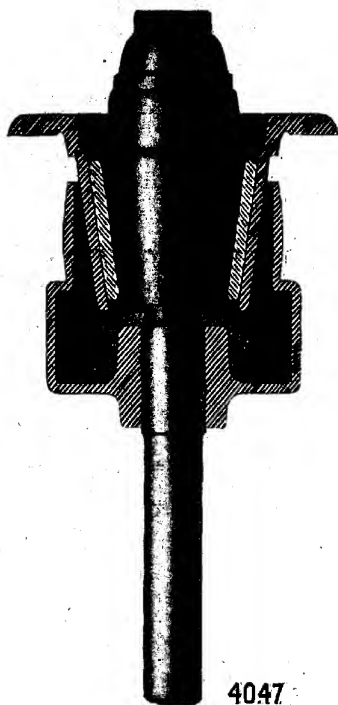
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PATENTEES AND SOLE MANUFACTURERS—

# **IMPROVED BALL-BEARING SPINDLE**

WITH

## **PATENT CONOIDAL BUFFER.**



4047

Code Word—"SINGLE."

(May be fitted to any existing Machine.)

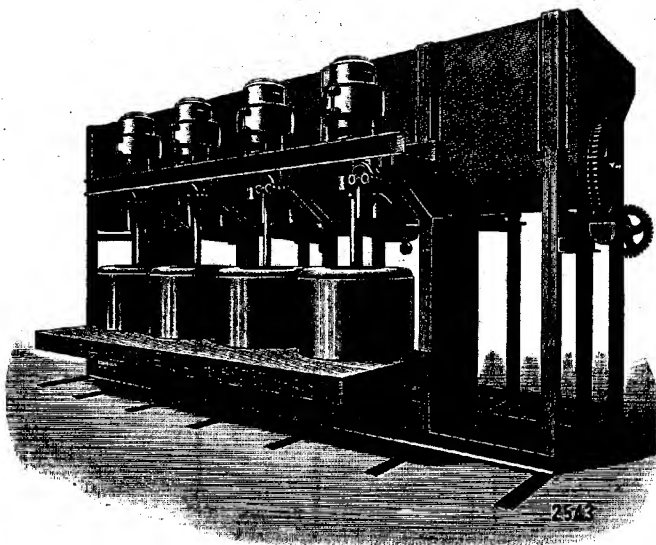
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**PATENTEES AND SOLE MANUFACTURERS—**

**WATSON, LAIDLAW & CO., Engineers, Glasgow.**

(5)

**PATENT STEEL FRAME**  
**AND**  
**PATENT ELECTRICALLY-DRIVEN "WESTON"**  
**CENTRIFUGAL MACHINES.**



Four Centrifugals, with **PATENT STEEL FRAMING** and **PATENT ELECTRIC-DRIVE**; Strike Mixer,  
 fitted with Scissors and Driving Gear.

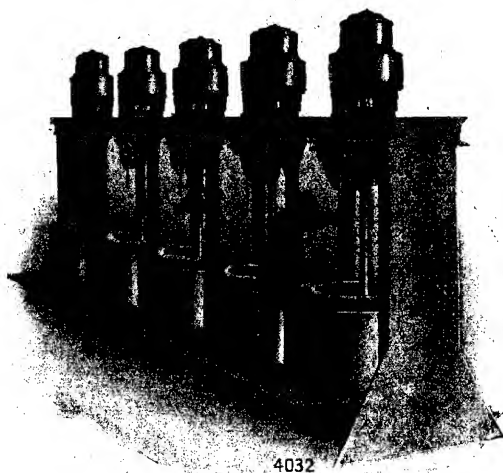
MADE WITH BASKETS—

30 ins. (762 m/m) diameter.	40 ins. (1000 m/m) diameter.
36 " (914 " ) "	42 " (1067 " ) "
48 ins. (1210 m/m) diameter.	

PATENTEES, AND SOLE MANUFACTURERS—

**WATSON, LAIDLAW & CO., Engineers, Glasgow.**  
 (6)

**PATENT STEEL FRAME**  
 AND  
**PATENT ELECTRICALLY-DRIVEN "WESTON"**  
**CENTRIFUGAL MACHINES.**



Five Centrifugals, with PATENT STEEL FRAMING and PATENT ELECTRIC-DRIVE.

(These Centrifugals are arranged for discharging through the floor.)

MADE WITH BASKETS—

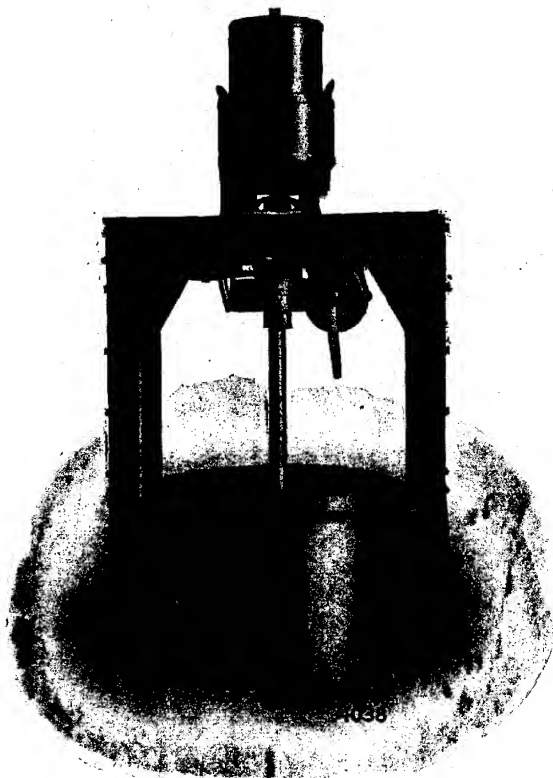
30 ins. (762 m/m) diameter.	40 ins. (1000 m/m) diameter.
36 " (914 " ) "	42 " (1067 " ) "
48 ins. (1220 m/m) diameter.	

PATENTEES AND SOLE MANUFACTURERS—

**WATSON, LAIDLAW & CO., Engineers, Glasgow.**  
 (7)



**PATENT STEEL FRAME**  
AND  
**PATENT ELECTRICALLY-DRIVEN "WESTON"**  
**CENTRIFUGAL MACHINES.**

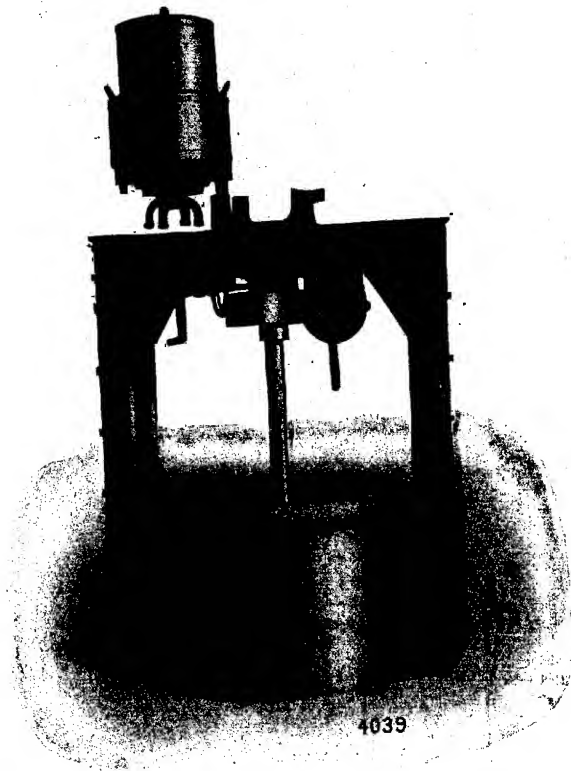


This illustration shows one of our Patent Improved Type of Electrically-driven Centrifugals.  
The illustration on page 9 shows the Motor disconnected.

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**PATENTEES AND SOLE MANUFACTURERS—**  
**WATSON, LAIDLAW & CO., Engineers, Glasgow.**  
(8)

**PATENT STEEL FRAME**  
**AND**  
**PATENT ELECTRICALLY-DRIVEN "WESTON"**  
**CENTRIFUGAL MACHINES.**

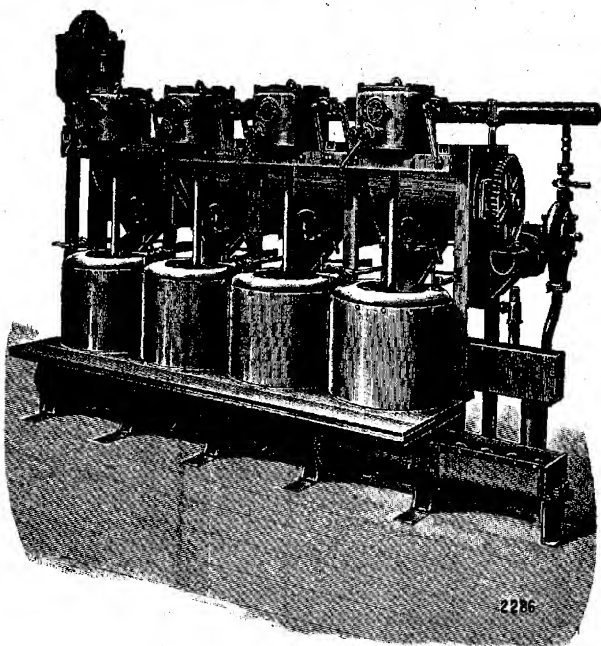


This illustration shows one of our Patent Improved Type of Electrically-driven Centrifugals with the Motor disconnected and swung round clear of the top of the Centrifuga Spindle, without any dismantling and without disturbing any electrical connection.

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**PATENTEES AND SOLE MANUFACTURERS—**  
**WATSON, LAIDLAW & CO., Engineers, Glasgow:**  
(9)

**PATENT STEEL FRAME**  
**AND**  
**PATENT WATER-DRIVEN "WESTON"**  
**CENTRIFUGAL MACHINES.**



Four Centrifugals, with PATENT STEEL FRAMING and PATENT WATER-DRIVE; Feeding Trough, fitted with Stirrers and Driving Gear; Pug Mill and Massacutts Elevator; Screw Conveyor; Steaming Cocks for inside and outside of Baskets.

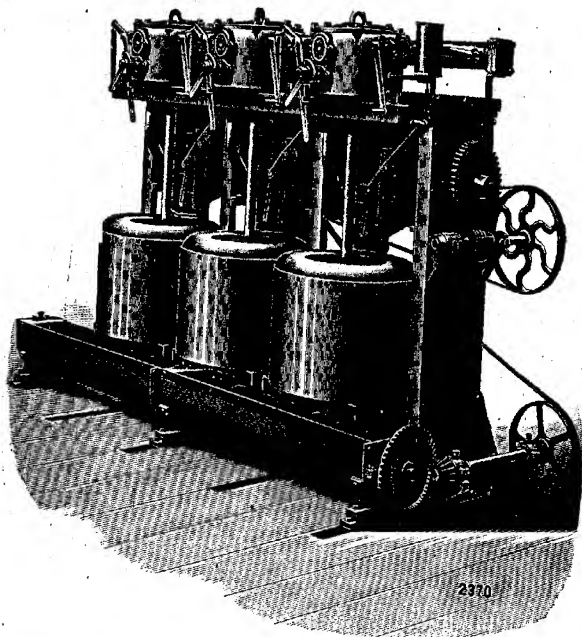
**MADE WITH BASKETS—**

30 ins. (762 m/m) diameter.	40 ins. (1000 m/m) diameter.
36 " (914 " ) " "	42 " (1067 " ) " "
48 ins. (1220 m/m) diameter.	

**PATENTEES AND SOLE MANUFACTURERS—**

**WATSON, LAIDLAW & CO., Engineers, Glasgow.**

**PATENT STEEL FRAME**  
**AND**  
**PATENT WATER-DRIVEN "WESTON"**  
**CENTRIFUGAL MACHINES.**



Three Centrifugals, with PATENT STEEL FRAMING and PATENT WATER-DRIVE; Feeding Trough,  
 fitted with Stirrers and Driving Gear; Screw Conveyor.

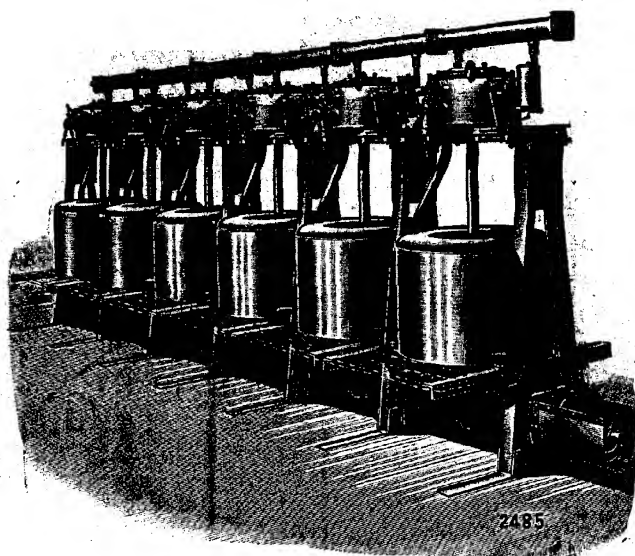
**MADE WITH BASKETS—**

30 ins. (762 m/m) diameter.	40 ins. (1000 m/m) diameter.
36 " (914 " ) "	42 " (1067 " ) "
48 ins. (1220 m/m) diameter.	

**PATENTEES AND SOLE MANUFACTURERS—**

**WATSON, LAIDLAW & CO., Engineers, Glasgow.**

**PATENT WATER-DRIVEN "WESTON"  
CENTRIFUGAL MACHINES WITH  
STEEL FRAMING.**



**Six Centrifugals, with PATENT STEEL FRAMING and PATENT WATER-DRIVE; Screw Conveyor.**

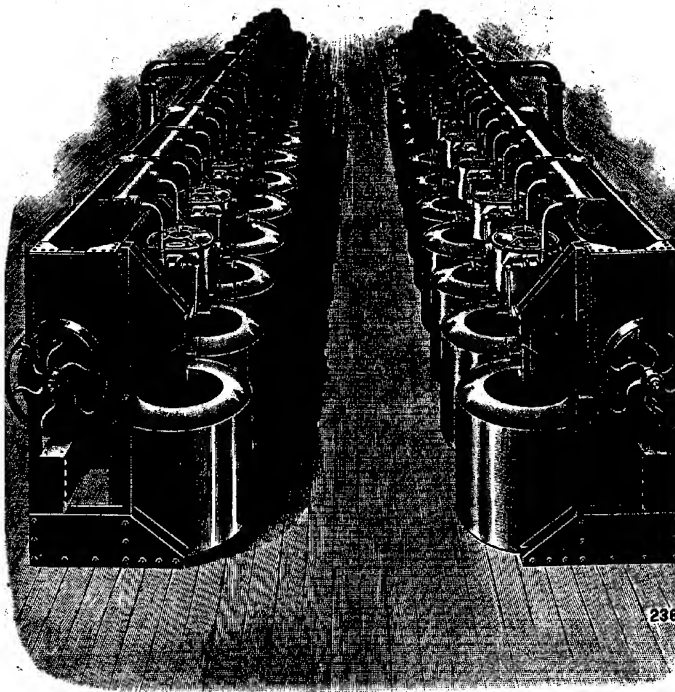
**MADE WITH BASKETS—**

30 ins. (762 m/m) diameter.	40 ins. (1000 m/m) diameter.
36 " (914 " ) "	42 " (1067 " ) "
48 ins. (1220 m/m) diameter.	

**PATENTEES AND SOLE MANUFACTURERS—**

**WATSON, LAIDLAW & CO., Engineers, Glasgow.**  
(12)

**PATENT STEEL FRAME**  
AND  
**PATENT WATER-DRIVEN "WESTON"**  
**CENTRIFUGAL MACHINES.**



Twenty-four Centrifugals, with PATENT STEEL FRAMING and PATENT WATER-DRIVE; Feeding Troughs, fitted with Stirrers and Driving Gear; Steaming Cocks and Pipes for inside and outside of Baskets.

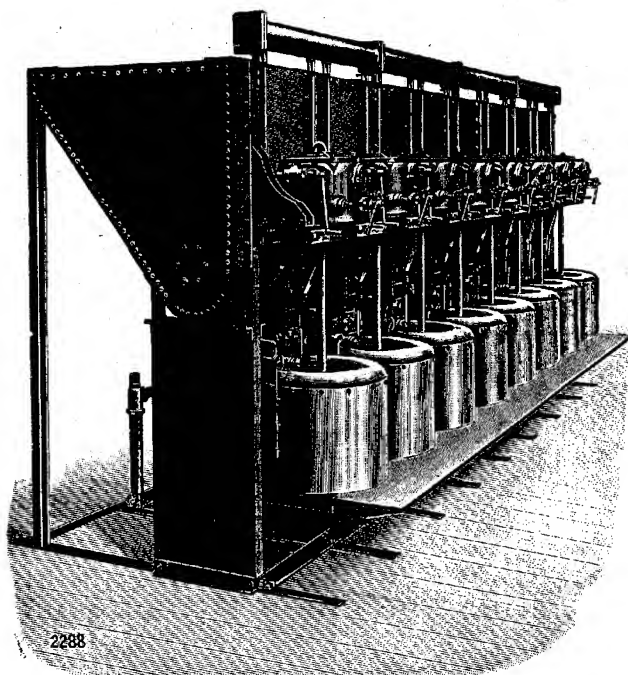
(These Centrifugals are arranged for discharging through the floor.)

MADE WITH BASKETS—

30 ins. (762 m/m) diameter.	40 ins. (1003 m/m) diameter.
36 " (914 " ) " "	42 " (1067 " ) " "
48 ins. (1220 m/m) diameter.	

PATENTEES AND SOLE MANUFACTURERS—  
**WATSON, LAIDLAW & CO., Engineers, Glasgow.**

**PATENT STEEL FRAME**  
**AND**  
**'PATENT WATER-DRIVEN "WESTON"**  
**CENTRIFUGAL MACHINES.**



**Eight Centrifugals, with PATENT STEEL FRAMING and PATENT WATER-DRIVE; Strike Mixer, fitted with Stirrers and Driving Gear; Steaming Cocks and Pipes for inside and outside of Baskets.**

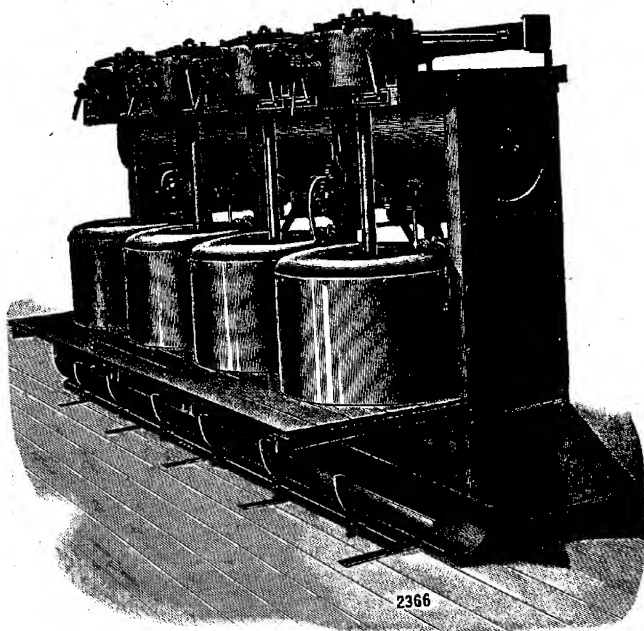
MADE WITH BASKETS—

30 ins. (762 m/m) diameter.	40 ins. (1000 m/m) diameter.
36 " (914 " ) "	42 " (1067 " ) "
48 ins. (1220 m/m) diameter.	

PATENTEES AND ~~SOLE~~ MANUFACTURERS—

**WATSON, LAIDLAW & CO., Engineers, Glasgow.**

**PATENT STEEL FRAME**  
 AND  
**PATENT WATER-DRIVEN "WESTON"**  
**CENTRIFUGAL MACHINES.**



Four Centrifugals, with PATENT STEEL FRAMING and PATENT WATER-DRIVE; Feeding Trough, fitted with Stirrers and Driving Gear; "Grasshopper" Conveyor; Steaming Cocks and Pipes for inside and outside of Baskets; Spraying Nozzles with Water Measures.

MADE WITH BASKETS—

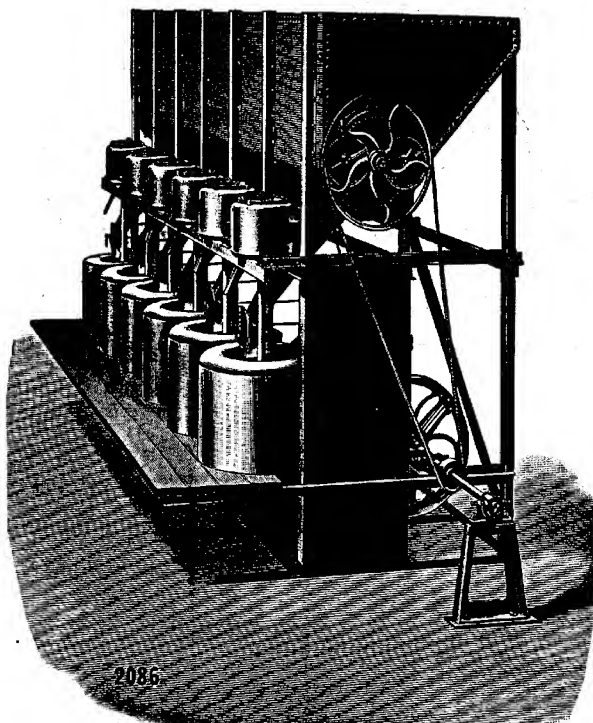
30 ins. (762 m/m) diameter.	40 ins. (1000 m/m) diameter.
36 " (914 " ) "	42 " (1067 " ) "
48 ins. (1220 m m) diameter.	

PATENTEES AND SOLE MANUFACTURERS—

**WATSON, LAIDLAW & CO., Engineers, Glasgow.**



**PATENT STEEL FRAME  
BELT-DRIVEN "WESTON" CENTRIFUGAL  
MACHINES.**



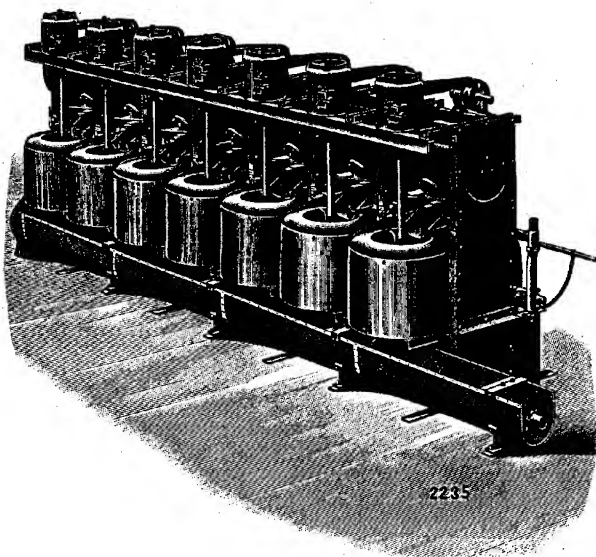
Six Centrifugals, with PATENT STEEL FRAMING and Belt-drive; Strike Mixer, fitted with Stirrers and Driving Gear.

MADE WITH BASKETS—

30 ins. (762 m/m) diameter.	40 ins. (1000 m/m) diameter.
36 " (914 " ) "	42 " (1067 " )
48 ins. (1220 m/m) diameter.	

PATENTEES AND SOLE MANUFACTURERS—  
WATSON, LAIDLAW & CO., Engineers, Glasgow.  
(16)

**PATENT STEEL FRAME  
BELT-DRIVEN "WESTON" CENTRIFUGAL  
MACHINES.**



Seven Centrifugals, with PATENT STEEL FRAMING and Belt-drive; Feeding Trough, fitted with Stirrers and Driving Gear; Screw Conveyor; Steaming Cocks and Pipes for inside and outside of Baskets.

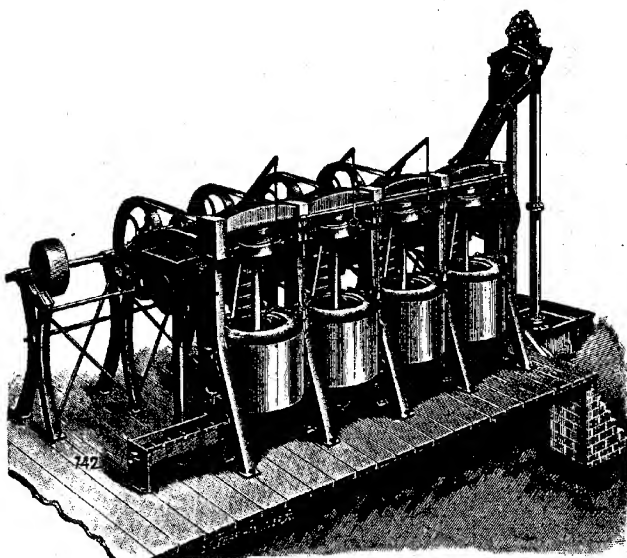
MADE WITH BASKETS—

30 ins. (762 m/m) diameter.	40 ins. (1000 m/m) diameter.
36 " (914 " ) "	42 " (1067 " ) "
• 48 ins. (1220 m/m) diameter.	

PATENTEES AND SOLE MANUFACTURERS—

**WATSON, LAIDLAW & CO., Engineers, Glasgow.**

**LAIDLAW'S**  
**PATENT SELF-BALANCING CENTRIFUGALS**  
 ("WESTON" TYPE).



Four Centrifugals, with PATENT INDEPENDENT FRAMING, Countershaft and Friction Pulleys, Feeding Trough, with Stirrers and Driving Gear; Pug Mill and Nasseculite Elevator; Dry Sugar Screw Conveyor; Steaming Cocks and Pipes for inside and outside of Baskets.

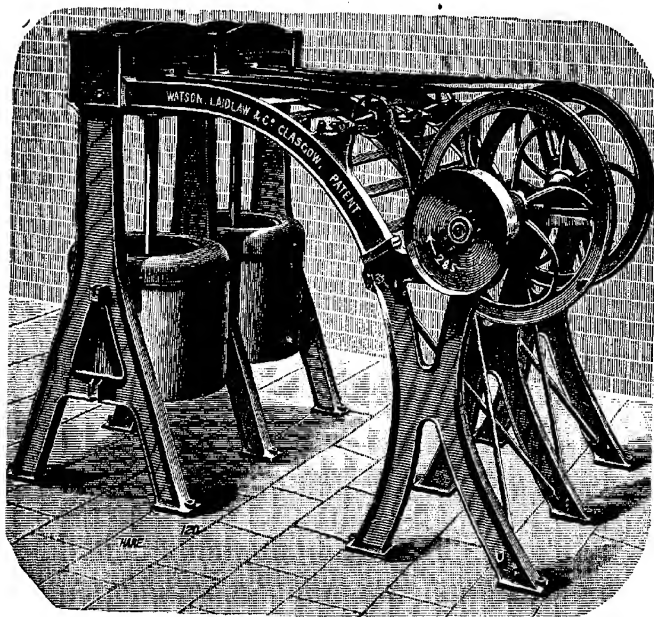
MADE WITH BASKETS—

30 ins. (762 $m/m$ ) diameter.	40 ins. (1000 $m/m$ ) diameter.
36 „ (914 „ ) „	42 „ (1067 „ )
48 ins. (122 $m/m$ ) diameter.	

PATENTEES AND SOLE MANUFACTURERS—

**WATSON, LAIDLAW & CO., Engineers, Glasgow.**

**LIDLAW'S**  
**PATENT SELF-BALANCING CENTRIFUGALS**  
 ("WESTON" TYPE).



Two Centrifugals, with PATENT INDEPENDENT FRAMING, Countershaft, Friction Pulleys and Driving Gear.

MADE WITH BASKETS—

30 ins. (762 m/m) diameter.	40 ins. (1000 m/m) diameter.
36 " (914 " ) " "	42 " (1067 " ) " "
48 ins. (1220 m/m) diameter.	

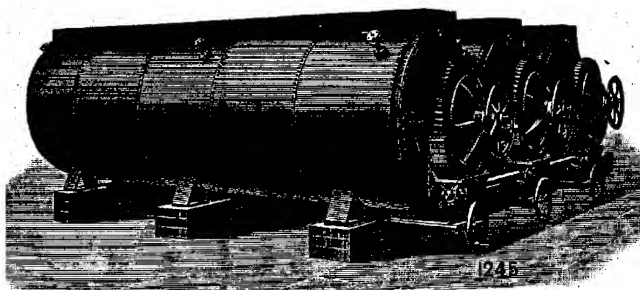
PATENTEES AND SOLE MANUFACTURERS—

WATSON, LIDLAW & CO., Engineers, Glasgow.  
 (19)

## **MASSEQUIE RECEIVERS**

FOR THE

## **CRYSTALLISATION OF SUGAR IN MOTION.**



The vessels shown are of the Semi-closed Type, Jacketed and fitted with Stirrers driven through friction gear. We also make vessels of open U shape section, and closed circular section, both with and without Jackets.

---

**PATENTEES AND SOLE MANUFACTURERS—**

**WATSON, LAIDLAW & CO., Engineers, Glasgow.**

(20)











